U.S. DEPARTMENT OF THE INTERIOR U.S. GEOLOGICAL SUEVEY

Modes of Occurrence of Trace Elements in Samples from a Coal Cleaning Plant

U.S. Geolocgical Survey

Curtis A. Palmer, Allan Kolker, Robert B. Finkelman, Kathleen C. Kolb,
Stanley J. Mroczkowski, Sharon S. Crowley, Harvey E. Belkin,
John H. Bullock, Jr., and Jerry M. Motooka

Open File Report 97-732

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Abstract

The overall objective of this project is to provide trace-element mode of occurrence information for CQ Inc. (CQ) under a Cooperative Research and Development Agreement (RADA) project entitled "Prediction of trace element removal from coal." This report provides semi-quantitative data on the modes of occurrence (chemical form) of 15 elements. Coals investigated include 4 plant feed coals from CQ Inc.'s coal cleaning facilities. Two of these feed coals are from the Pittsburgh bed, one from West Virginia and one from Pennsylvania. The other two feed coals are bituminous coal samples taken from the Kittanning* and Freeport* coal zones of Pennsylvania. In addition to these feed coals, trace-element modes of occurrence were determined for the cleaned coal product and for two intermediate fractions, the feed and product from the froth flotation step. Characterization techniques include scanning electron microscopy, electron microprobe analysis, and selective leaching procedures. Microprobe data show that many pyrite grains in the four feed coals have measurable concentrations of As, Cu, Ni, and Zn, generally in the 0-0.05 weight percent range. Concentrations of other chalcophile elements (Se, Cd, Co) in pyrite are generally below the detection limit of 0.01 weight percent. Pyrites in the Freeport and Kittanning coal zones show arsenic enrichment; maximum As contents exceeding 1.0 weight percent were determined for pyrite in the flotation concentrate fraction of the Freeport coal zone. Taken together, microprobe, selective leaching data, and other techniques indicate that As is primarily associated with pyrite, and a substantial portion of Cr is associated with illite. The other 12 elements studied have multiple modes of occurrence.

^{*} In this report we will refer to these samples as coming from the Kittanning or Freeport coal zones.

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Introduction

The United States Geological Survey (USGS) has been collaborating with CQ Inc. to better understand the influence of the modes of occurrence of trace elements on their behavior during coal cleaning. Information from this collaborative project will be used to develop models for predicting the response of potentially toxic trace elements in commercial coal cleaning procedures. In the present report we have determined the modes of occurrence of 15 elements in four additional coals. We have also determined the modes of occurrence in the final cleaned products and two intermediate materials: the froth flotation feed and the cleaned material from the froth flotation. The unique contribution of this research is that it relates the behavior of elements in a commercial coal cleaning procedure to their modes of occurrence in the coal. This work should lead to models for forcasting the cleaning behavior of elements in our Nation's coal resources.

In the present study, CQ collected four samples of bituminous coal, three from Pennsylvania and one from West Virginia. Two of these coals are from the Pittsburgh coal bed; one is from the Kittanning coal zone and one from the Freeport coal zone. Each of these coals were cleaned in CQ's pilot scale coal cleaning facility. Figure 1 shows a flow diagram of the steps involved with the coal cleaning process and the 4 samples for each coal for which we determined trace-element modes of occurrence. Splits of the original feed coals, froth flotation feed, froth flotation cleaned fraction, and final cleaned product were sent to the USGS for trace element modes of occurrence analysis. Project goals include (1) developing quantitative trace-element mode of occurrence information and developing mechanisms for trace-element removal by coal cleaning, to aid in realizing the full potential of HAPs control technologies; (2) reducing this knowledge to engineering practice, and (3) assembling the information in a form that can be used by industry on a routine basis. In support of this effort, the USGS has analyzed 16 coal samples or related coal fractions utilizing the techniques described below, to provide information necessary to achieve a better understanding of toxic element behavior.

All of the samples have been processed by a selective leaching procedure, a technique for approximating elemental modes of occurrence, using differing combinations of solvents. Splits of the coal have been leached with ammonium acetate, hydrochloric acid, hydrofluoric acid, and nitric acid, according to the methods originally developed at the USGS by Finkelman et al. (1990). Results of these leaching tests provide essential information on chemical bonding of the elements present. Elements that are leached by hydrofluoric acid are generally associated with silicates, those that are leached by nitric acid generally occur in sulfides, and those that are leached by hydrochloric acid generally occur in carbonates and mono-sulfides. Ammonium acetate will leach elements that are weakly attached to exchangeable organic sites or are water soluble.

A split of each coal sample was ashed using a low temperature ashing device. This procedure causes oxidation of the coal at temperatures of less than 200° C, producing a residue of generally unaltered minerals. This low temperature ash residue was analyzed for semi-quantitative mineralogy by X-ray diffraction analysis.

The above procedures provide indirect evidence, or approximations of the modes of

occurrence of trace elements in coal. These procedures are complemented by direct approaches such as manual scanning electron microscopy (SEM), with energy dispersive analysis (EDX) and microprobe studies of polished pellets of unashed coal samples and coal cleaning products.

Methods

The sequential selective leaching procedure used in this study is similar to one described by Palmer et al. (1993) which was modified from that of Finkelman et al. (1990). Duplicate 5g samples were sequentially leached with 35 ml each of 1N ammonium acetate (CH₃COONH₄), 3N hydrochloric acid (HCl), concentrated hydrofluoric acid (HF; 48%) and 2N (1:7) nitric acid (HNO₃) in 50 ml polypropylene tubes. Each tube was shaken for 18 hrs using a Burrell¹ wrist action shaker. Because gas can form during some of the leaching procedures it is necessary to enclose each tube in double polyethylene bags, each closed with plastic-coated wire straps. The bags allow gas to escape, but prevent the release of liquid. Approximately 0.5 g of residual solid was removed from each tube for instrumental neutron activation analysis (INAA) and cold vapor atomic absorption (CVAA) analysis for mercury. The solutions were saved for inductively-coupled argon plasma (ICP) analysis and inductively-coupled argon plasma mass-spectroscopy (ICP-MS) analysis.

SEM and Microprobe

1 - Coal pellet casting and polishing

The pellet formation procedure follows the ASTM D2797-85 (ASTM 1997) technique for anthracite and bituminous coal. The casting procedure impregnates, under pressure, approximately 7-8 grams of crushed sample with potting epoxy. The resultant mold is cured overnight at 60° C. A label is incorporated with the sample.

The pellet block is ground and polished in accordance with ASTM D2797-85 (ASTM 1997) standards. The epoxy-coal pellet is ground with a 15 μ m diamond platen and 600-grit SiC paper until flat and smooth. Rough polishing is done with 1 μ m alumina and final polishing is completed with 0.06 μ m colloidal silica. Ultrasonic cleaning between and after the various steps insures a final product free of extraneous abrasive material.

Two pellets were prepared from each sample. A wafer approximately 2 mm thick was cut from the polished end of the pellet cylinder with a thin, slow-speed diamond saw; the wafer was then carbon coated for SEM and microprobe analysis.

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2 - Scanning electron microscope analysis.

A JEOL-840 scanning electron microscope equipped with a Princeton Gamma-Tech. energy-dispersive X-ray analytical system and an ETEC Autoscan with Kevex EDX were used for SEM examination of project coals. Mineral identifications are assigned on the basis of morphology, and major-element composition of grains. Both secondary electron imaging (SEI) and back-scattered electron imaging (BSE) modes were used in coal sample characterization. The BSE mode is especially sensitive to variation in mean atomic number, and is useful for showing within-grain compositional variation. By optimizing the BSE image, the presence of trace phases containing elements with high-atomic number can be revealed if the concentration of the trace elements are high enough. Samples were examined initially to obtain an overall view of the phases present, as with a petrographic microscope. This initial examination was followed by a series of overlapping traverses in which the relative abundance of the phases was assessed. EDX analysis provides information on elements having concentrations at roughly the tenths-of-percent level or greater based on subsequent wavelength dispersive spectroscopy analysis. operating conditions for SEM analysis were: accelerating potential of 10-30 kV, magnifications of ~50->10,000 times and working distances ranging from 25 or 39 mm (JEOL) and 15 to 20 mm (ETEC). The results of the SEM analyses can be found in Appendix I.

Photomicrographic scanning electron microscopy preceded electron microprobe analysis. SEI or BSE images taken at low magnification were used as a guide to locate phases of interest for microprobe analysis. SEM images taken at higher magnifications provide maps of the points analyzed. Images at higher magnifications commonly reveal the presence of pits, interstices or other imperfections in mineral grains that may not be visible in reflected light microscopy. SEI/BSI mapping enables us to avoid such features that would adversely affect the sums of the quantitative analyses of mineral grains using the microprobe.

3 - Electron microprobe analysis.

A fully-automated, 5 spectrometer instrument (JEOL JXA 8900L Superprobe) was used to quantitatively determine element concentrations in sulfides and clays by the wavelength-dispersive technique. In our microprobe work on sulfides, pyrite grains were selected to represent each of the morphologies observed. Grains selected for analysis were well-polished and generally lacked pits and other imperfections that would adversely affect the analysis. In addition, analyses totaling <95% were rejected. The following elements were measured: Fe, S, As, Ni, Cu, Zn, Se, Co, and Cd. Natural and synthetic standards were used. Beam current used was 3.0 x10⁻⁸ amps; accelerating voltage was 20 KeV. The probe diameter was set as a focused beam; the actual working diameter was about 3-5 micrometers. This limited the acceptable minimum grain size of minerals for quality analysis to about 10 micrometers. In this study, the minimum detection limit for the microprobe was about 100 ppm for each of the elements analyzed in pyrite, using counting

times of 60 seconds for peak and 30 seconds for background for minor or trace elements present. Trace elements can be detected in sulfides at the 100 ppm level; however, counting statistics at this concentration have a large uncertainty. In the probe analysis, we attempted to detect compositional differences among different pyrite morphologies. Microprobe data collected are shown in Appendix II. In addition to quantitative analyses, the JEOL 8900L was used to produce color maps of elemental distribution in project sulfides. Similar microprobe procedures were used for the analysis of clay minerals (Appendix II), with the following exceptions: beam current used was 2.0 x 10⁻⁸ amps, accelerating voltage was 15 KeV, and estimated detection limit is about 200 ppm.

X-Ray diffraction analysis

To obtain semi-quantitative information on the minerals present in the study coals, samples of low- temperature (<200° C) ash were pressed into pellets and X-rayed using an automated diffractometer. The X-ray signals were scanned over an interval from 4° to 60° 2θ. Counts were collected at 0.5 seconds per step. The data were processed using a computer program for semi-quantitative mineral analysis by X-ray diffraction (Hosterman and Dulong, 1985).

Results and Discussion

SEM and Microprobe Analysis

SEM analysis indicates the presence of the major minerals illite, kaolinite, quartz, and pyrite in all 16 samples analyzed (Appendix I). In addition to these four minerals, major amounts of calcite were found in all samples except those from the Freeport coal zone. In addition, major to minor amounts of iron oxides were found in all samples except the plant feed for the Pennsylvania Pittsburgh coal, the cleaned coal product from the West Virginia Pittsburgh coal, and the plant feed for the Kittanning coal zone. Other minerals were found in minor and trace amounts (Appendix I).

1 - Microprobe Analysis of Fe-sulfides

Microprobe data for most pyrite grains indicate trace-element concentrations that are at or near the detection limit of ~100 ppm (Appendix IIA). Of the seven trace elements were determined (Se, Cu, Ni, As, Zn, Cd, and Co), only Cu, As, Ni, and Zn are commonly present at measurable levels.

As and Ni concentrations in pyrite grains from each of the coals studied are shown in Figure 2. As and Ni were chosen because they are the HAP's elements most commonly present in concentrations above the detection limit for the samples in this study. In the Freeport coal zone samples, all sample splits contain pyrite grains having arsenic contents that exceed approach or exceed 0.50 weight percent (Fig. 2a). The highest As values (approaching 1.25 wt. %) were determined for cell filling pyrite grains in the flotation

concentrate fraction. These data are consistent with previous results for the Upper Freeport coal, in which maximum As contents exceeding 1.5 weight percent have been reported (Minkin et al., 1984; Ruppert et al. 1992). Despite the trend towards high As in the Freeport coal zone, it is clear that the bulk of the As and Ni data, for pyrite, clusters around the origin in Figure 2a. The data for pyrite in the cleaned product show the most pronounced clustering at the origin (Fig. 2a).

Some of the pyrite grains in the Kittanning coal zone samples contain high (>0.5 wt. %) arsenic values. Unlike the Freeport coal, data for the Kittanning coal zone samples indicate that high arsenic pyrite is most common in the cleaned product. As-rich pyrite grains include subhedral, round, composite and euhedral forms. Data for this fraction also suggests that As enrichment in many pyrite grains is correlated with Ni enrichment (to about 0.10 wt. % Ni; Fig 2b). This apparent correlation between As and Ni was not found in any other fraction of the four coals examined (Fig. 2).

Trace element distributions for pyrite in the two Pittsburgh coal samples are similar, each primarily showing minor variation in Ni (below detection limit to > 0.05 wt. %) that is independent of As. Each Pittsburgh coal contains isolated pyrite grains that approach (PA cleat filling) or exceed (frambod from WV) 0.25 weight percent As (Fig. 2c-d).

Arsenic contents of pyrite grains are plotted vs. their maximum dimensions in Figure 3. Pyrite grains in each fraction of the Freeport coal zone samples, spanning a size range from 15 to 750 micrometers, show similar trends towards arsenic enrichment (Fig. 3a). There is no apparent correlation between arsenic content and grain size. In the Kittanning coal, the most As-rich grains are examined in the cleaned product. All of the Kittaning coal zone fractions show a restricted size interval (mostly < 100 micrometers) as a result of the cleaning process and the apparent lack of considerably larger pyrite grains in the plant feed fraction (Fig. 3b). However, processing of the plant feed fractions appears to result in significant reduction of pyrite grain size for the Freeport coal zone and Pittsburgh (PA) coal samples. In each of these coals, pyrite grains that are > 500 micrometers are present in the original feed fractions (Fig. 3a, 3c). The plots are not meant to represent the complete grain-size distribution of the pyrite grains, but the analyses are useful in illustrating the compositional variation of the pyrite grains. In addition to quantitative analysis, the microprobe was used to show within-grain trace-element variation in selected pyrite grains. Figure 4 shows a cluster of arsenic-rich (maximum point analysis = 0.83 wt. % As) pyrite framboids in the cleaned coal fraction of the Kittanning coal zone. The As map at the lower left of Figure 4 shows that the framboids have As-rich cores that are mantled by progressively As-poorer compositions. The Ni map at the upper right of Figure 4 shows similar enrichment at the centers of grains. The figure illustrates compositional changes that took place during the growth and agglomeration of the framboids. Other Kittanning coal zone framboids do not show As enrichment.

2 - Microprobe Analysis of Clay Minerals

Microprobe analyses of the clay mineral illite are given in Appendix IIB. The illite analyses are limited to the plant feed sample for each of the coals because this fraction contains large illites that are not finely disseminated. Because only a portion of the illite is

removed in the cleaning process, compositions of illites obtained from the plant feed coals should be similar to those in other fractions of each coal.

 ${\rm Cr_2O_3}$ concentrations of illites in the plant feed samples range from below the detection limit (about 0.02 wt. %, equivalent to about 137 ppm Cr), to 0.04 weight percent (equivalent to about 274 ppm Cr). Values at or near the detection limit are subject to a large uncertainty. Nevertheless, these data indicate that illite is an important source of Cr in the coals in this study.

The illites also show large major-element variations, likely due to the presence of mixed-layer clays and finely disseminated quartz, making it difficult to analyze a truly representative illite. Illite analyses in Appendix IIB have sums that are less than 100 percent because they contain structural water in these clays.

Semi-Quantitative Mineralogy of Low-Temperature Ash

Table 1 gives semi-quantitative estimates of mineralogy based on X-ray diffraction analysis (XRD) of low-temperature ash (LTA) of all 16 samples About 10 to 20 percent of the ash of each of the LTA samples consists of quartz, 30 to 65 percent of the LTA is kaolinite and 10 to 30 percent of the LTA is illite. The LTA for the Pittsburgh coals and related sub-samples contain 5 to 15 percent pyrite. The LTA for the other coals and related sub-samples contains only trace amounts to 5 percent pyrite. Calcite makes up a trace to 5 percent of the LTA for the Pittsburgh and the Kittanning coal zone and related subsamples, but calcite was not detected in any of the Freeport coal zone LTA samples or subsamples. This finding is in agreement with SEM results in which calcite was also not detected in the Freeport coal. The LTA of the Kittanning coal zone and the Freeport coal zone feed and froth flotation feed LTA each contains 15 percent chlorite. The LTA from the cleaned froth flotation sub-sample contains 10 percent chlorite. Discrete grains of chlorite were not identified by SEM (Appendix I). Feldspar, bassinite, gibbsite, hematite and apatite were found in minor to trace quantities in several coals. Feldspar, present in trace amounts according to XRD, was not observed by SEM.

Leaching Experiments

Leaching experiments were completed in duplicate for the four program feed coals and their sub-samples. The resulting leachate solutions and solid residues were submitted for chemical analysis. ICP-AES and ICP-MS analyses (for leachate solutions) and INAA analyses (for solid residues) were obtained. Chemical data for the leachates and residues were processed to derive the percentages of each element leached by each of the four leaching agents. The calculated percentages were then used as an indirect estimate of the mode of occurrence of specific trace elements in the coals. By comparing data of the residual fractions with data from the solutions, we estimate a relative error of up to ± 25 percent for the percent leached data.

Percent leached data for each trace element studied in the plant feed (PF), froth feed (FF), froth clean (FC) and plant clean (PC) fractions of each of the four coals are shown in Figures 5 through 19.

1 - Arsenic

Forty-five to eighty-five percent of the As is leached by HNO₃ in 14 of the 16 coals and sub-samples (Fig. 5) indicating an association with pyrite (Table 3). Extraction of As account for 90 to 95 percent of the total arsenic in most coals. The two exceptions are the Pittsburgh plant clean coals. In these two samples more than 65 percent of the arsenic remained after the entire leaching procedure, with only 0 and 5 percent of the total arsenic leached by HNO₃ in the Pittsburgh PA and WV cleaned coals, respectively. examination of the residues of the Pittsburgh cleaned coals revealed that significant amounts of pyrite were still present after leaching. This probably accounts for most of the un-leached arsenic in these samples. The residue samples were re-leached with nitric acid and most of the unleached iron was leached during this second leach. SEM examination of this leach revealed only trace amounts of pyrite. This data suggest that if organically bound arsenic could only exist in very small amounts if at all. The reason for the presence of pyrite in the original residual is not clear, but the subsequent removal of pyrite suggests that the rate of dissolution of pyrite may be kinetically controlled. The remaining 14 samples had less than 10 percent of the arsenic remaining after leaching. Arsenates and silicates (probably clays) account for 10 to 45 percent of the arsenic.

Arsenic in all samples was partially soluble (5 to 40%) in HCI. HCI-soluble arsenic may be present as arsenates in the coal, or due to the formation of arsenates by oxidation of pyrite. It is also possible that there are HCI-soluble arsenic-bearing sulfides present. Microprobe data confirms the presence of arsenic in pyrite but also shows its distribution to be very heterogeneous.

2- Mercury

The behavior of mercury is similar to that of arsenic. However, its low concentrations and volatility make mercury much more difficult to measure. Mercury was determined by cold vapor atomic absorption on a split of the solid residue. In three of the sixteen samples, no mercury was leached. These samples include both of the Pittsburgh plant-cleaned coals and the froth concentrate from the Pennsylvania Pittsburgh coal. In the case of the froth concentrate, the original (raw) concentration of mercury was at the detection limit of 0.02 ppm and therefore the concentration in all leached fractions was below the detection limit. Hg concentrations in the Pittsburgh cleaned coals were also near the detection limit but are as high as other coals for which we have leaching data. In the case of the Pittsburgh coals, the lack of leached Hg is probably due in part to residual un-leached pyrite. However, there are some indications that Hg behaves slightly differently than other elements associated with pyrite and some Hg may be absorbed on the surfaces of organic rich particles (Table 3). Further work is needed to resolve this issue. The froth flotation fraction of the Kittanning coal zone has only 30 percent Hg leached suggesting that it too may have

significant quantities of organic associated or shielded Hg. Figure 6 shows that in all other samples, 50 to 75 percent of the Hg is leached by HNO₃ indicating 50 to 75 % of the Hg is in pyrite (Table 3). In addition, 15 to 30 percent of Hg is leached by HCl in three of the West Virginia Pittsburgh coal sub-samples. The HCl-leachable Hg may be associated with oxidized pyrite or HCl soluble sulfides.

3 - Chromium

Unlike As and Hg, Cr is mostly associated with silicates. Figure 7 shows that 20 to 55 percent of the Cr is leached by HF. Microprobe analysis of illite grains in the study coals indicates Cr_2O_3 concentrations ranging from 0 to 0.04 weight percent. Minor amounts of Cr were also leached by HCl and HNO $_3$ in several of the samples. HNO $_3$ -leached Cr is probably associated with sulfides. Thirty to eighty percent of the Cr is unleached and remains in the HNO $_3$ leached residues. SEM examination did not reveal any illite in the residual fractions. The remaining Cr is therefore most likely due to organically associated Cr or sub-microscopic shielded illite grains or submicroscopic insoluble spinels such as chromite. The 5 to 10 percent HCl soluble Cr may be due to oxyhydroxides as reported by Huggins and Huffman (1996).

4 - Selenium

Figure 8 shows 25 to 65 percent of the Se remains in the HNO₃-leached residue. This is most likely due to an organic Se association. Some of the Se is also associated with sulfides. Although 10 to 70 percent of the Se is leached by HNO₃, oxidation of organics by HNO₃ may release some organically-bound Se, which would then be indistinguishable from Se released from the sulfides. Microprobe data of individual pyrite grains indicates an average Se concentration at or below the detection limit of 0.01 weight percent. No Se was leached from the Pittsburgh (PA) plant cleaned coal by any solvents and only 10% of the Se was leached from the Pittsburgh (WV) coal by HNO₃. Again, the residual un-leached pyrite in these samples probably accounts for a significant portion of the Se. Some Se in several of the samples was leached by ammonium acetate, possibly indicating the presence of exchangeable or water-soluble Se compounds. Lead selenide (PbSe) was detected by SEM in the plant clean fraction of the Kittanning coal zone, which may account for some of the HCI-leachable Se in that sample.

5 - Nickel

Table 3 suggests four modes of occurrence for Ni. Significant amounts of Ni are leached by HCl, HF, and HNO₃ (Fig. 9). Because up to about 65 percent of the Ni in our samples remains un-leached by HNO₃, Ni in this form represents the forth mode of occurrence and may be organically associated, or in small particles shielded by organics. Ni is generally present at the 0.01 to 0.05 weight percent level in the pyrites, but Ni concentrations approaching 0.30 weight percent have been found (Fig. 2). The relatively

high analytical errors of Ni values determined by INAA at or near their detection limits, especially in the solid residues, contributes to the overall uncertainty.

6 - Cobalt

The behavior and therefore the modes of occurrence of cobalt is similar to that of Ni (Fig. 10; Table 3). HCl was the most effective solvent, but like Ni, substantial amounts of Co were leached by HF and HNO₃. Co has also been detected in some pyrite grains. Up to 38 percent of the Co remains un-leached by HNO₃, therefore some of the Co may be organically associated, or in small particles shielded by organics. Although the concentration of Co is lower than that of Ni, INAA is much more sensitive for Co, making the analytical uncertainties significantly lower for Co.

7 - Antimony

The behavior of antimony (Sb), is also similar to Co and Ni (Fig. 11; Table 3). In addition to a sulfide association, some Sb is associated with the silicates, as evidenced by significant amounts of Sb leached by HF in all samples; some Sb probably exists as oxides as well (Table 1; HCl leachable). Up to 60 percent of the Sb remains in the HNO₃ leached solid residues suggesting that some of the Sb may be organically associated or in small mineral grains shielded by organics.

8 - Lead

Galena is a common accessory mineral in coals (Finkelman, 1994). Twenty to fifty-five percent of the lead is leached by HCl and HNO₃ (Fig. 12), consistent with galena as the major source of Pb in these coals. SEM analysis confirmed the presence of galena in all feed coal samples. The wide range of un-leached Pb may be due to shielding of submicroscopic galena grains or an organic association. Pb was not determined in the residues because Pb cannot be determined using the INAA procedures employed in this study. Lead selenide (PbSe) was detected by microprobe in the Kittanning coal zone feed coal sample which may account for some HCl leachable Pb in that sample. In addition, 5 to 25 percent of the Pb was leached by HF, indicating an association with silicates.

9 - Cadmium

The data for Cd are limited. Cd was not detected in four of the whole coal samples, and the concentration of Cd in the other 12 samples was at or near the detection limit. The leaching data for the four samples that had whole coal values below the detection limit (Fig. 13) represents the lower limits of the leached values, based on the data from the leached solutions and the detection limits of Cd in the whole coal samples. As Figure 13 illustrates, Cd is primarily soluble in HCl and HNO₃. These data are consistent with a sphalerite (ZnS) association. Sphalerite is a common accessory mineral in coal, and was detected by SEM in all four of the coal samples (Appendix I). Cd concentrations are below the detection limit in all of the pyrites analyzed by microprobe (Appendix II). The leaching behavior of Zn (Fig.

14), is not similar to Cd, as would be expected if the Cd and Zn both occurred only in sphalerite. However, the different leaching behavior of Zn may be due to Zn contamination from oxidized mining or handling equipment as indicated by high amounts of ammonium-acetate-leached Zn in several of the samples. Five to thirty-five percent of the Cd was leached by HF, indicating an association with silicates. Up to 90 percent of the Cd remained in the HNO₃ residues. This may be due to shielding of sub-microscopic sphalerite grains by organics. In the case of the Pittsburgh plant clean coals, there was no Cd leached by HNO₃. It is possible that in these samples, sphalerite is being shielded in the same manner as the pyrite. However, no un-leached residual sphalerite grains were detected by SEM in the leached residues.

10 - Beryllium

Beryllium is generally associated with the silicates. Be is not detectable by microprobe or SEM due to its low atomic number. It is also not detected in the residues (by INAA). Data plotted in Figure 15 shows that 30 to 70 percent of Be is leached by HF. In addition, 5-10 percent of the Be is HCl leachable. This may be due to the presence of Be oxides (Table 3). Up to 65 percent of the beryllium remained in the HNO₃-leached residues, most likely as a result of organic association.

11 - Manganese

Manganese is primarily HCI-leachable, and is mostly associated with carbonates (Fig. 16). Significant amounts of Mn are also leachable by HF and HNO₃ suggesting associations with silicates and sulfides, especially in the Freeport coal zone (Table 3). Microprobe analysis of illite grains in the study feed coals indicates Mn concentrations ranging from 0 to 0.07 weight percent (Appendix IIB). The lack of HNO₃-leachable Mn in the cleaned coals from the Pittsburgh seam may be to the difficulty of dissolving pyrite in this sample. Up to 30 percent of the Mn remained in the HNO₃ leached residues. This is most likely due to shielded carbonates. One important point to note is that up to 35 percent of the Mn in these samples was ammonium-acetate leachable (Fig. 16). This is probably contamination from oxidized Mn-bearing steel mining and grinding equipment as has been previously reported by Finkelman et al. (1984). Mn in the residues was not determined by INAA due to the relatively short half-life (2.5 hours) of ⁵⁶Mn.

12 - Uranium

Uranium is mostly leached by HF indicating an association with silicates (Fig. 17, Table 3). However, significant amounts of U are also leached by HCl and HNO₃. Uranium oxides are soluble in HNO₃. Thirty to seventy-five percent of the U remained in the HNO₃ leached residue, suggesting an organic association or the presence of insoluble phases such as zircon (Appendix I).

13 - Thorium

The leaching pattern for Th varies slightly among the program coals. Thorium in the Pittsburgh coals is leached significantly by HCl and HNO₃, and to a lesser extent by HF. The Kittanning and Freeport coal zone samples, however, have the greatest leaching contribution from HNO₃. The HNO₃-leached Th may be attributable to phosphates (e.g. monazite). Twenty-five to eighty percent of the Th remained in the HNO₃ leached residues. This is probably due to insoluble phases such as zircon (Table 3).

14 - Iron

Iron is a major element in pyrite, which also contains many of the HAP's elements. We have therefore included iron in many of our tables. The leaching data for the program coal samples are presented in Figure 19. Although pyrite is the major mode of occurrence for Fe in the Pittsburgh coals and the Freeport coal zone samples, the Kittanning coal zone samples have significant amounts of HCl and HF leachable material indicating that iron in this coal is also in the silicates, sulfates, oxides and/or carbonates. Figure 19 clearly illustrates the lack of HNO₃ leached Fe in the Pittsburgh cleaned coals. None of the Fe in the Pennsylvania coal and only 5 percent of the Fe in the West Virginia coal was leached by HNO₃, Again, this is most likely due to the presence of residual un-leached pyrite in these two samples. Subsequent leaching of these residues removed 95 percent of the remaining iron and all but trace amounts of the pyrite.

SEM Examination of Leached Residues

To assess the effectiveness of the leaching process, solid residues remaining after leaching were examined by SEM. A total of 8 samples, representing the Plant Feed and Plant Cleaned coal fractions of each program coal were examined. In most of the residues, a TiO₂ phase, probably rutile, is the most common mineral. TiO₂ was originally present as a minor- or trace phase, and is relatively insoluble in the combination of reagents employed in the leaching process. Isolated grains of other insoluble phases, such as zircon, and minor amounts of more abundant phases, such as pyrite, illite, and quartz, were observed locally. Two exceptions are the two Pittsburgh cleaned coals. In each of these leached residues, significant amounts of undigested or partially digested pyrite were observed. This observation accounts for the anomalous leaching results for As (only 0-5% As leached by nitric acid) and other pyritically associated elements, compared to larger percentages in the other fractions of the Pittsburgh coals.

Semi-quantitative Modes of Occurrence

Combining all of the information from the leaching experiments, the microprobe and scanning electron microscopy, X-ray diffraction analysis and the various chemical analyses, as well as geochemical characteristics of each element, semi-quantitative assessments of each trace-element's modes of occurrence have been determined. Table 3 includes

determinations of the percent of each element present partitioned among three or four major phases or minerals, for 15 elements. In cases where there is significant direct evidence, the exact form of the mineral is given, such as As in pyrite or Cr in illite. In cases where there is strong geochemical evidence and strong indirect evidence, classes of minerals are given, such as sulfides, silicates, oxides or arsenates. In some cases a descriptor is used, such as HCI-soluble sulfides to distinguish these species from other sulfides, such as pyrite.

Conclusion

The USGS has analyzed the four coals (Two from the Pittsburgh coal seam (PA and WV), one from the Kittanning coal zone (PA), and one from the Freeport coal zone (PA)) and their related intermediate and final cleaned products. Modes of occurrence of trace elements in coal were determined by: (1) leaching experiments, (2) SEM analysis, and (3) microprobe analysis. Semi-quantitative results for modes of occurrence of fifteen elements have been provided. Chemical analysis of whole coals by several techniques (ICP-AES. ICP-MS, cold vapor atomic absorption, hydride generation), has been completed. Additional chemical analysis on individual pyrite and illite grains were determine by microprobe analysis. Finally, mineralogical information on each of the four coals and related sub-samples has been provided. These results mark an important advance in quantifying the modes of occurrence of elements in coal. This type of information may prove useful in developing better procedures for coal cleaning and provide information needed by power companies for coal selection. Additional work needs to be done to determine why pyrite was not completely removed in the nitric acid step for some coals. Further work on the residuals could differentiate between organically associated elements and elements contained in fine particles encased in the organic matrix.

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Tables

- 1. Semi-Quantitative Ash Mineralogy
- 2. Analytical Values for Whole Coals
- 3. Control Samples
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 - B. ICP-AES
 - C. ICP-MS
 - D. Cold Vapor (Hg) and Hydride Generation (Se) Atomic Absorption
- 4. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products.

Table 1. Semi-Quantitative Ash Mineralogy. All values in percent*

Bassinite ND ND minor trace	trace minor ND trace	Q Q	ND ND trace ND
Hematite ND trace trace ND	ND ND trace ND	ND trace trace ND	ND trace ND
Gibbsite ND trace ND ND ND	ND krace ND	trace ND ND ND ND ND ND ND	ND trace trace
Apatite trace minor ND trace	ND N	trace tra N trace	trace trace ND ND
Eeldspar trace ND trace trace	trace ND minor minor	trace trace ND trace	trace trace ND trace
Chlorite ND ND ND		15 15trace inortrace ND	15 15 10 trace
Calcite minor 5 minor trace	5 ND minor	ND trace tracemir ND	
Pyrite 5 10 10 5 5	5 5 5 5 7 5 7	trace trace minor trace	minor minor 5
20 20 15 10	15 15 20 20	30 20 20 20	8888
Kaolinite 50 50 60 60	50 55 55 55	30 40 65 70	30 35 35 50
Quartz 20 15 10	10 10 11	20 10 10	20 20 10
Sample PF401 FF401 FC401 PC401	PF701 FF701 FC701 PC701	PF801 FF801 FC801 PC801	PC901 FF901 FC901 PC901

*Analyst F.B. Dulong minor=less than 5 % greater than trace trace= appears to be present

Table 2. Analytical Values for Whole Coals

Plant Concentrate - INAA Plant Concentrate - Denver Techniques	Froth Concentrate - INAA Froth Concentrate - Denver Techniques	Froth Feed - INAA Froth Feed - Denver Techniques	Plant Feed - INAA Plant Feed - INAA Dup Plant Feed - Denver Techniques	96032701 Pittsburgh Coal (WV)	Plant Concentrate - INAA Plant Concentrate - INAA Dup Plant Concentrate - Denver Techniques	Froth Concentrate - INAA Froth Concentrate - Denver Techniques	Froth Feed - INAA Froth Feed - Denver Techniques	Plant Feed - INAA Plant Feed - Denver Techniques	96052401 Pittsburgh Coal (PA)	Element units:
5.4 5.4	4.4 5.5	6.7 7.5	12.0 11.8 15		3.2 3.8	4.6 5.6	7.1 9.2	9.6 12		As ppm
N D 0.08	0.06	ND 0.07	ND ND 0.15		0.06	ND 0.02	0.06	0.13		Hg
11.6 14	11.7 13	18.9 16	16.5 17 18		11.3 11.3 13	11.8 12	15.6 16	19.2 20		Cr
1.4 1.7	0.9 1.8	0.9 1.2	1.7 1.9 2.8		0.9 1.0 1.0	1.1 0.50	1.0	1.4 2.5		Se
^ 13 5.9	6.2 6.2	< 21 8.3	< 23 18 11		8.9 8.9	9.9 9.9	< 21 12	16 14		ppm <u>N</u> :
2.18 2.4	2.45 2.6	3.2 3.4	ω ω ω το 4 το		2.7 2.65 2.9	3.0 3.1	3.9 4.2	4.6 5.0		Co
0.27 0.2	0.29 0.29	0.32 0.3	0.35 0.35 0.77		0.28 0.27 0.34	0.40 0.45	0.45 0.55	0.51 0.5		Sb
4.3	ND 3.4	4.6	0.7 6.7		3.5 0 0	3.9	4.9	8.2		Pb ppm
ND 0.07	0.06	0.1	^0.1 0.1		0.00 ND	0.06	0. 1 0. 1	0. ND		ppm
4.7 5.5	19 18	70 78	19 17 18		7.4 9	10.5 12	17 17	17 20		Zn
ND 0.7	ND 0.74	0.8 0.8	0.7 0.7		0.81	ND 0.74	ND 0.7	0.9		Be ppm
16 16	18 ND	40 40	35 N N		8. N N N N N N N N N N N N N N N N N N N	13 N	33 N	26 26		M n ppm
1.60 1.9	1.28 1.4	1.58 1.8	2.5 2.48 2.5		1.54 1.55 1.5	1.37 1.4	1.89 2.2	2.8 2.9		Th
0.45 0.53	0.44 0.55	0.60 0.62	0.80 0.76 0.96		0.45 0.58 0.52	0.51 0.57	0.61 0.64	0.8 4 0.96		mdd O
0.87 0.92	0.90 0.91	1.80 1.8	1.94 1.94 2.0		0.55 0.57 0.52	0.69 0.69	1.64 1.4	1.11		Fe

Denver Techniques: ICP: Be, Co, Cr, Mn, Ni, Th, Zn, Fe. ICP-MS: As, Cd, Pb, Sb, U. Hydride Generation Atomic Absorption: Se. Cold Vapor Atomic Absorption: Hg. ND = not determined

Table 2. Analytical Values for Whole Coals (continued)

0.18 17 0.95 13	20.2 ND 16.0	Froth Feed - INAA 22.0 ND 25.0 < 1.1 15 6.0 Froth Feed - INAA Dup 23.8 ND 26.8 0.8 18 5.5 Froth Feed - Denver Techniques 28 0.21 25 0.96 16 5.8	Plant Feed - INAA 43 ND 31 1.1 26 7.5 Plant Feed - Denver Techniques 55 0.50 36 1.2 22 7.7	96032901 Freeport Coal Zone (PA)	Plant Concentrate - INAA 4.7 ND 22.3 4.2 22 6.9 Plant Concentrate - Denver Techniques 7.2 0.16 30 5.2 22 7.5	Froth Concentrate - INAA	Froth Feed - INAA 8.2 ND 29.9 4.0 22 7.5 Froth Feed - INAA Dup 7.7 ND 29.1 4.0 24 7.5 Froth Feed - Denver Techniques 11 0.14 32 6.0 21 7.2	Plant Feed - INAA 12.9 ND 47 6.1 28 10.0 Plant Feed - Denver Techniques 18 0.25 60 5.3 33 11	96032801 Kittanning Coal Zone (PA)	Element As Hg Cr Se Ni Co units: ppm ppm ppm ppm ppm
0.48 ND	0.57 ND 0.51 5.4	0.53 ND 0.51 ND 0.74 6.6	0.62 ND 0.8 11		1.88 ND 2.2 13	1.54 ND 1.52 ND 1.9 14	1.54 ND 1.54 ND 1.8 16	1.76 ND 2.0 25		Sb Pb ppm ppm
		ND 38 ND 38 0.3 82								Cd Zn ppm ppm
1.2	0.96	7 <u>0</u> 0	2 2 2		1.9 1.9	1.5 ND	7 D D	2 N 20		
9 N 8 D	ND 17	ND ND 53	38 38		ND 7.4	1 N N	39 39	ND 57		M n
1.87 2.0	1.76 1.8	2.66 2.7 2.8	4.4 5.0		4.0 4.0	3.0 3.06 2.7	4.20 4.1 3.9	8.1 6.7		Th
0 6 6 6	0.61 0.63	0.95 0.8 0.78	1.13 1.1		1.7 2.0	1.3.5	1.9	2.6 2.5		ppm U
0.88 0.86	0.98 0.78	2.49 2.45 2.3	1.68 1.8		0.282 0.32	0.355 0.335 0.37	1.13 1.10 1.1	1.03 1.2		% Fe

Denver Techniques: ICP: Be, Co, Cr, Mn, Ni, Th, Zn, Fe. ICP-MS: As, Cd, Pb, Sb, U. Hydride Generation Atomic Absorption: Se. Cold Vapor Atomic Absorption: Hg. ND = not determined

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Arsenic 96052401 Pittsburgh Coal (PA) Plant Feed Froth Feed Froth Concentrate Cleaned Coal	Pyrite 55% 555% 65%	Arsenates 30% 30% 25% 20%	Silicates 10% 10% 10% 5%	Organic or Shielded Minerals* 5% 10%
Cleaned Coal 96032701 Pittsburgh Coal (WV)	65%	20%	5%	10%
Plant Feed	65%	15%	10%	10%
Froth Feed	55%	25%	10%	10%
Froth Concentrate	45%	30%	15%	10%
Cleaned Coal	60%	25%	5%	10%
96032801 Kittanning Coal Zone (PA)	Ď			
Plant Feed	60%	20%	10%	10%
Froth Feed	65%	20%	10%	5%
Froth Concentrate	65%	15%	10%	10%
Cleaned Coal	85%	5%	5%	5%
96032901 Freeport Coal Zone (PA)	•			
Plant Feed		10%	5%	5%
Froth Feed	80%	10%	5%	5%
Froth Concentrate	75%	10%	5%	10%
Cleaned Coal	75%	10%	5%	10%

^{*}Likely shielded pyrite.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Mercury		HCI Soluble		Organic or
	Pyrite	Sulfides	Silicates	Shielded Minerals*
96052401 Pittsburgh Coal (PA)				
Plant Feed	75%	0%	5%	20%
Froth Feed	70%	5%	0%	25%
Froth Concentrate	0%	0%	0%	100%**
Cleaned Coal	75%	0%	0%	25%
96032701 Pittsburgh Coal (WV)				
Plant Feed	50%	15%	0%	35%
Froth Feed	50%	30%	10%	10%
Froth Concentrate	50%	15%	5%	30%
Cleaned Coal	50%	0%	0%	50%
96032801 Kittanning Coal Zone (PA)				
Plant Feed	55%	0%	0%	45%
Froth Feed	70%	0%	0%	30%
Froth Concentrate	30%	0%	0%	70%
Cleaned Coal	50%	0%	0%	50%
96032901 Freeport Coal Zone (PA)				
Plant Feed	75%	0%	5%	20%
Froth Feed	75%	0%	0%	25%
Froth Concentrate	70%	0%	5%	25%
Cleaned Coal	60%	0%	0%	40%

^{*}Likely ½ organic association and ½ shielded pyrite; however, many fractions may be below the detection limit, and, as a result, may overestimate this value.

^{**}Original material at detection limit for this element; all fractions likely below detection limit. (See text for discussion.)

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Chromium	IIIite	Sulfides	Oxyhydroxides(?)	Organic or Shielded Minerals*
96052401 Pittsburgh Coal (PA)			•	
Plant Feed	40%	5%	5%	50%
Froth Feed	30%	0%	10%	60%
Froth Concentrate	30%	0%	5%	65%
Cleaned Coal	20%	0%	0%	80%
96032701 Pittsburgh Coal (WV)				
Plant Feed	40%	0%	5%	55%
Froth Feed	30%	0%	10%	60%
Froth Concentrate	30%	0%	5%	65%
Cleaned Coal	25%	0%	0%	75%
96032801 Kittanning Coal Zone (PA)				
Plant Feed	50%	15%	5%	30%
Froth Feed	35%	10%	5%	50%
Froth Concentrate	25%	5%	0%	70%
Cleaned Coal	30%	5%	0%	65%
96032901 Freeport Coal Zone (PA)				
Plant Feed	55%	10%	5%	30%
Froth Feed	45%	5%	10%	40%
Froth Concentrate	30%	5%	5%	60%
Cleaned Coal	25%	5%	5%	65%

^{*}Likely organic association.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Selenium 96052401 Pittsburgh Coal (PA) Plant Feed Froth Feed Froth Concentrate Cleaned Coal 96032701 Pittsburgh Coal (WV) Plant Feed Froth Feed Froth Concentrate Cleaned Coal	Pyrite 60% 60% 25% 60% 70% 50%	Silicates 0% 0% 15% 0% 0% 5%	Accessory and Mono-Sulfides 10% 10% 10% 0% 5% 10% 5% 10%	Organic or Shielded Minerals* 30% 50% 40% 25% 30% 40% 80%
Cleaned Coal	10%	0%	10%	80%
96032801 Kittanning Coal Zone (PA) Plant Feed	40%	5%	5%	50%
Froth Feed	30%	5%	10%	55%
Cleaned Coal	40%	0%	0%	60%
96032901 Freeport Coal Zone (PA) Plant Feed	60%	0%	0%	40%
Froth Feed Froth Concentrate	40% 35%	0% 0%	0% 0%	60% 65%
Cleaned Coal	30%	10%	0%	60%

^{*}Likely organic association.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Nickel Su	Sulfides	Ni oxides	Silicates	Organic or Shielded Minerals*
96052401 Pittsburgh Coal (PA)				
Plant Feed	20%	30%	15%	35%
Froth Feed	30%	25%	20%	25%
Froth Concentrate	30%	30%	20%	20%
Cleaned Coal	30%	20%	15%	35%
96032701 Pittsburgh Coal (WV)				
Plant Feed	35%	40%	20%	5%
Froth Feed	35%	40%	20%	5%
Froth Concentrate	40%	40%	20%	0%
Cleaned Coal	40%	30%	20%	10%
96032801 Kittanning Coal Zone (PA)				
Plant Feed	35%	10%	50%	5%
Froth Feed	25%	15%	25%	35%
Froth Concentrate	10%	10%	15%	65%
Cleaned Coal	20%	10%	25%	45%
96032901 Freeport Coal Zone (PA)				
Plant Feed	30%	20%	15%	35%
Froth Feed	25%	15%	20%	40%
Froth Concentrate	30%	15%	20%	35%
Cleaned Coal	35%	15%	15%	35%

^{*}Likely organic association.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Cobalt 96052401 Pittsburgh Coal (PA) Plant Feed Froth Feed Froth Concentrate Cleaned Coal 96032701 Pittsburgh Coal (WV) Plant Feed	Sulfides 10% 15% 10% 15%	HCI Soluble 40% 35% 20%	Silicates 10% 15% 10% 10%	Organic or Shielded Minerals* 40% 35% 50% 55%
96032701 Pittsburgh Coal (WV) Plant Feed	30%	30%	15%	25%
Froth Feed Froth Concentrate	10% 10%	30% 30%	15% 10%	45% 50%
Cleaned Coal	20%	15%	10%	55%
96032801 Kittanning Coal Zone(PA)				
Plant Feed Froth Feed	20% 25%	15% 15%	25% 15%	40% 45%
Froth Concentrate	20%	10%	10%	60%
Cleaned Coal	20%	10%	10%	60%
96032901 Freeport Coal Zone (PA) Plant Feed	20%	25%	15%	40%
Froth Feed	20%	15%	20%	45%
Froth Concentrate	15% 20%	20% 15%	15%	50%
Cleaned Coal	20%	15%	10%	55%

^{*}Likely organic association.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

lfides	Silicates	Oxides	Organic or Shielded Minerals*
		,	
20%	15%	25%	40%
15%	20%	25%	40%
15%	20%	25%	40%
15%	15%	20%	50%
25%	15%	10%	50%
20%	20%	25%	35%
20%	20%	20%	40%
15%	15%	20%	50%
15%	20%	5%	60%
10%	20%	15%	55%
10%	25%	5%	60%
10%	25%	5%	60%
30%	20%	10%	40%
15%	20%	15%	50%
15%	20%	15%	50%
25%	15%	10%	50%
	Sulfides 20% 15% 15% 15% 25% 20% 10% 10% 10% 10% 10% 10% 15% 25%	20% 15% 20% 15% 20% 15% 15% 20% 15% 15% 15% 15% 15% 20% 15% 20% 20% 15% 20% 10% 25% 20% 10% 25% 20% 15% 25% 20% 15% 20% 15% 20% 15% 20% 15% 20% 15% 20% 15% 20% 20% 15% 20% 20% 20% 20% 20% 20% 20% 20% 20% 20	Sii

^{*}Likely organic association.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Lead 96052401 Pittsburgh Coal (PA) Plant Feed Froth Feed Froth Concentrate	Galena 45% 55%	Silicates 10% 20%	Organic or Shielded Minerals* 45% 25%
Cleaned Coal 96032701 Pittsburgh Coal (WV)	20%	20%	60%
Plant Feed Froth Feed	60% 70%	10% 15%	30% 15%
Froth Concentrate	55%	25%	20%
Cleaned Coal	20%	10%	70%
96032801 Kittanning Coal Zone (PA)	5	g	
Plant Feed Froth Feed	70% 65%	0%	30% 35%
Froth Concentrate	40%	10%	50%
Cleaned Coal	45%	15%	40%
96032901 Freeport Coal Zone (PA) Plant Feed	70%	10%	20%
Froth Feed	45%	5%	50%
Cleaned Coal	45%	5%	50%

^{*}Likely shielded galena.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

*Likely shielded sphalerite.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Zinc Sph 96052401 Pittsburgh Coal (PA) Plant Feed Froth Feed	Sphalerite 65% 80%	Silicates 15% 15%	Organic or Shielded Minerals* 20% 5%
Froth Concentrate Cleaned Coal	60% 20%	5% 35%	35% 45%
96032701 Pittsburgh Coal (WV) Plant Feed	70%	15%	15%
Froth Feed	50%	10%	40%
Froth Concentrate	60%	15%	35%
Cleaned Coal	25%	50%	25%
96032801 Kittanning Coal Zone (PA)			
Plant Feed	50%	40%	10%
Froth Feed	30%	5%	65%
Froth Concentrate	45%	15%	40%
Cleaned Coal	45%	30%	25%
96032901 Freeport Coal Zone (PA)			
Plant Feed	55%	30%	15%
Froth Feed	50%	15%	35%
Froth Concentrate	70%	20%	10%
Cleaned Coal	35%	25%	40%

^{*}Likely shielded sphalerite.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Beryllium			Organic or
	Silicates	Oxides	Shielded Minerals*
96052401 Pittsburgh Coal (PA)			
Plant Feed	70%	10%	20%
Froth Feed	55%	5%	40%
Froth Concentrate	50%	5%	45%
Cleaned Coal	55%	5%	40%
96032701 Pittsburgh Coal (WV)			
Plant Feed	70%	10%	20%
Froth Feed	55%	5%	40%
Froth Concentrate	55%	5%	40%
Cleaned Coal	65%	5%	30%
96032801 Kittanning Coal Zone (PA)			
Plant Feed	55%	10%	35%
Froth Feed	45%	10%	45%
Froth Concentrate	30%	5%	65%
Cleaned Coal	55%	5%	40%
96032901 Freeport Coal Zone (PA)			
Plant Feed	55%	10%	35%
Froth Feed	35%	5%	60%
Froth Concentrate	50%	5%	45%
Cleaned Coal	30%	5%	65%

*Likely organic association.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Manganese Carbo 96052401 Pittsburgh Coal (PA)	Carbonates	Sulfides	Silicates	NH4-Acetate Soluble*	Organic or Shielded Minerals**
Plant Feed	45%	25%	10%	20%	0
Froth Feed	20%	15%	25%	20%	20%
Froth Concentrate	30%	25%	15%	25%	(D
Cleaned Coal	20%	25%	20%	30%	45
96032701 Pittsburgh Coal (WV)					
Plant Feed	40%	40%	10%	10%	
Froth Feed	40%	15%	25%	20%	0%
Froth Concentrate	30%	25%	20%	20%	
Cleaned Coal	20%	25%	15%	35%	
96032801 Kittanning Coal Zone (PA)					
Plant Feed	45%	10%	25%	10%	10%
Froth Feed	45%	5%	25%	25%	
Froth Concentrate	40%	0%	30%	30%	
Cleaned Coal	25%	0%	35%	35%	
96032901 Freeport Coal Zone (PA)					
Plant Feed	30%	30%	30%	0%	_
Froth Feed	25%	10%	40%	5%	2
Froth Concentrate	25%	25%	40%	5%	
Cleaned Coal	10%	60%	25%	0%	5%

^{*}NH4-Acetate soluble; likely contaminants from stainless steel (mining equipment).

^{**}Likely shielded carbonates.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Uranium				Organic or
SI	Silicates	HCI Soluble	Oxides	Shielded Minerals*
96052401 Pittsburgh Coal (PA)				
Plant Feed	25%	15%	10%	50%
Froth Feed	25%	10%	10%	55%
Froth Concentrate	15%	10%	5%	70%
Cleaned Coal	20%	5%	5%	70%
96032701 Pittsburgh Coal (WV)				
Plant Feed	25%	15%	10%	50%
Froth Feed	30%	10%	5%	55%
Froth Concentrate	25%	5%	5%	65%
Cleaned Coal	25%	0%	5%	70%
96032801 Kittanning Coal Zone (PA)				
Plant Feed	40%	5%	25%	30%
Froth Feed	25%	5%	10%	60%
Froth Concentrate	15%	0%	10%	75%
Cleaned Coal	30%	5%	10%	55%
96032901 Freeport Coal Zone (PA)				
Plant Feed	40%	5%	20%	35%
Froth Feed	35%	0%	15%	50%
Froth Concentrate	25%	5%	10%	60%
Cleaned Coal	15%	5%	10%	70%

^{*}Likely ½ organic association and ½ insoluble phases (e.g. zircon).

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

60%	Froth Concentrate 30% 50% 10%	40%	35%	60%	60%	Froth Feed 25% 40% 15%	25%	80%	75%	Froth Feed 15% 60% 10%	50%	96032701 Pittsburgh Coal (WV)	70%	70%	Froth Feed 10% 50% 15%		96052401 Pittsburgh Coal (PA)	Phosphates Insoluble* Silicates	
10%	10%	20%	15%	5%	10%	15%	10%	10%	10%	10%	10%		5%	10%	15%	10%		Silicates	
5%	10%	5%	5%	5%	5%	20%	15%	0%	5%	15%	20%		10%	10%	25%	30%		HCI Soluble	

*Insoluble phases such as zircon.

Table 3. Modes of Occurrence of Trace Elements in Feed Coals and Intermediate and Final Coal Cleaning Products (continued) (Best estimates based on SEM, microprobe, XRD, and leaching data.)

Iron			Carbonates	Organic or
96052401 Pittsburgh Coal (PA)	Pyrite	Silicates	and Sulfates	Snieided Minerais"
Plant Feed	65%	10%	15%	10%
Froth Feed	35%	35%	30%	0%
Froth Concentrate	65%	20%	15%	0%
Cleaned Coal	70%	10%	10%	10%
96032701 Pittsburgh Coal (WV)				
Plant Feed	85%	10%	5%	0%
Froth Feed	40%	35%	20%	5%
Froth Concentrate	55%	20%	20%	5%
Cleaned Coal	75%	5%	10%	10%
96032801 Kittanning Coal Zone (PA)				
Plant Feed	25%	40%	25%	10%
Froth Feed	20%	40%	35%	5%
Froth Concentrate	40%	40%	15%	5%
Cleaned Coal	55%	35%	10%	0%
96032901 Freeport Coal Zone (PA)				
Plant Feed	70%	5%	15%	10%
Froth Feed	25%	45%	25%	5%
Froth Concentrate	40%	35%	15%	10%
Cleaned Coal	80%	10%	5%	5%

^{*}Likely shielded pyrite.

Figures

- 1. Flow diagram of the steps of the coal cleaning process and the 4 samples for each coal for which we determined trace-element modes of occurrence.
- 2. Plots of nickel vs. arsenic for pyrites in program coals and related subsamples: 2a) Freeport coal zone; 2b) Kittanning coal zone; 2c) Pittsburgh (PA) coal; 2d) Pittsburgh (WV) coal.
- 3. Plots of maximum dimension vs. arsenic content for pyrites in program coals and related sub-samples: 3a) Freeport coal zone; 3b) Kittanning coal zone; 3c) Pittsburgh (PA) coal; 3d) Pittsburgh (WV) coal.
- 4. Semi-quantitative elemental map of Se, Co, As, and Ni distribution in a ~150 micrometer cluster of pyrite framboids in the 51 Plant Cleaned Coal fraction of the Kittanning coal zone. Figure shows high-As framboid centers that have been concentrically overgrown by pyrite with lower As concentrations. Ni distribution (upper right) mimics As variation. Figure also shows Co and Se present at uniformly low concentrations. Pixel size is 0.5 micrometers; width of field-of-view is 175 x 175 micrometers (350 x 350 pixels). Dwell time at each pixel is 500 msec. Maximum As concentration, as determined by previous quantitative analysis, is about 0.80 weight percent.
- 5. Determination of percent arsenic leached from feed coals and intermediate and final coal cleaning products.
- 6. Determination of percent mercury leached from feed coals and intermediate and final coal cleaning products.
- 7. Determination of percent chromium leached from feed coals and intermediate and final coal cleaning products.
- 8. Determination of percent selenium leached from feed coals and intermediate and final coal cleaning products.
- 9. Determination of percent nickel leached from feed coals and intermediate and final coal cleaning products.
- 10. Determination of percent cobalt leached from feed coals and intermediate and final coal cleaning products.
- 11. Determination of percent antimony leached from feed coals and intermediate and final coal cleaning products.

- 12. Determination of percent lead leached from feed coals and intermediate and final coal cleaning products.
- 13. Determination of percent cadmium leached from feed coals and intermediate and final coal cleaning products.
- 14. Determination of percent zinc leached from feed coals and intermediate and final coal cleaning products.
- 15. Determination of percent beryllium leached from feed coals and intermediate and final coal cleaning products.
- 16. Determination of percent manganese leached from feed coals and intermediate and final coal cleaning products.
- 17. Determination of percent uranium leached from feed coals and intermediate and final coal cleaning products.
- 18. Determination of percent thorium leached from feed coals and intermediate and final coal cleaning products.
- 19. Determination of percent iron leached from feed coals and intermediate and final coal cleaning products.

Figure 1 Flow Diagram for Coal Cleaning Process



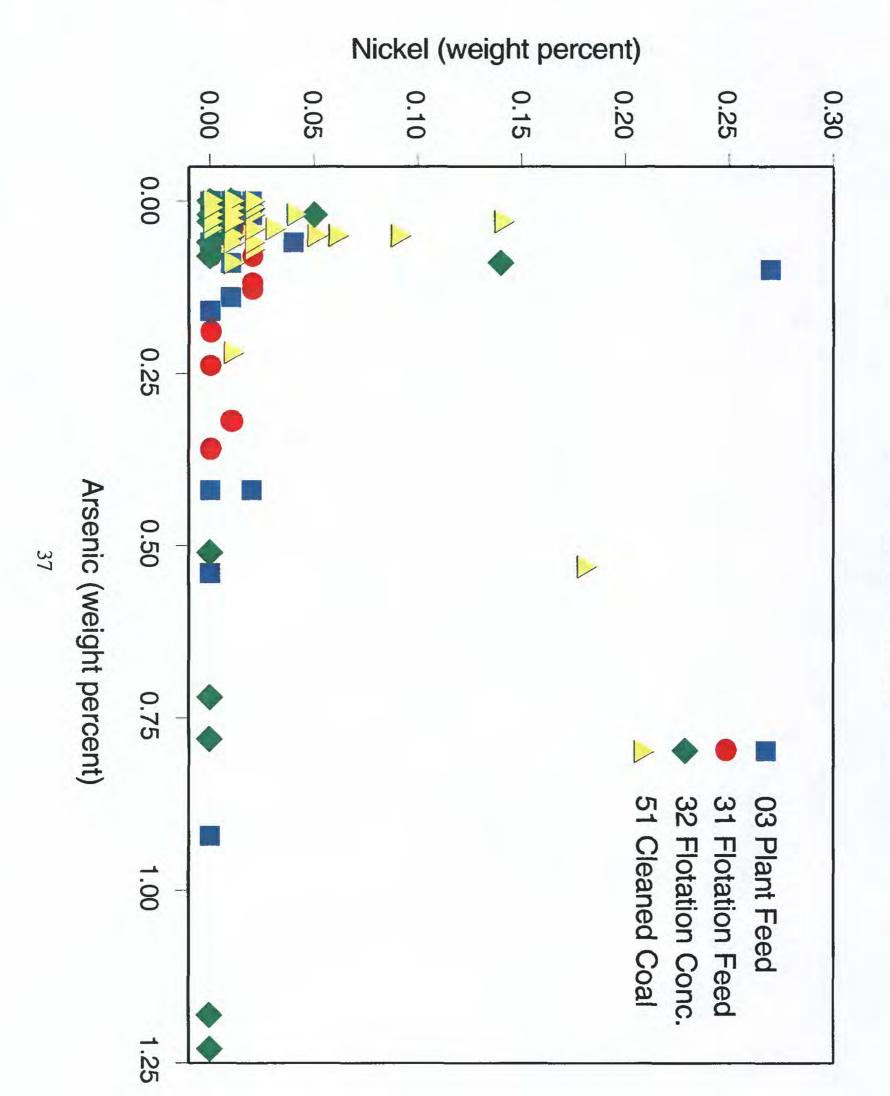


Figure 2a
Freeport (PA)

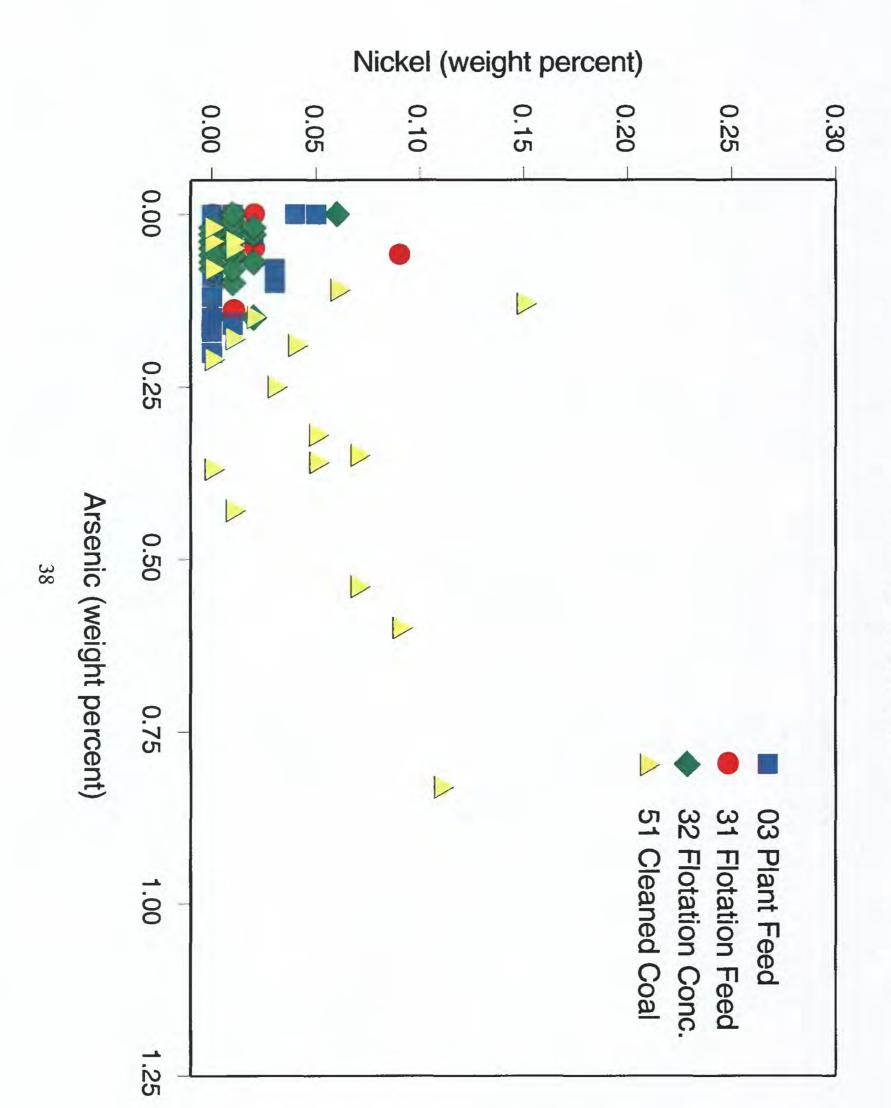


Figure 2b Kittanning (PA)

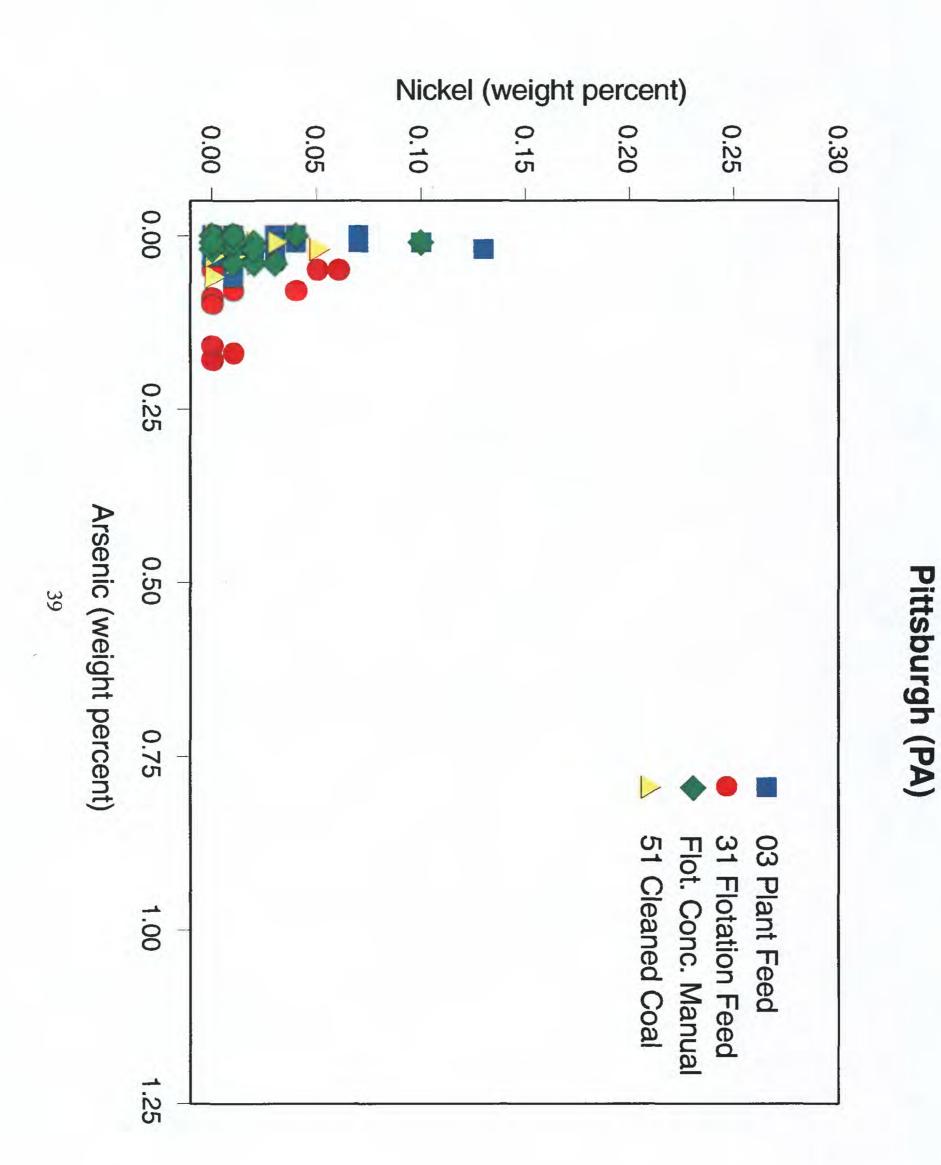
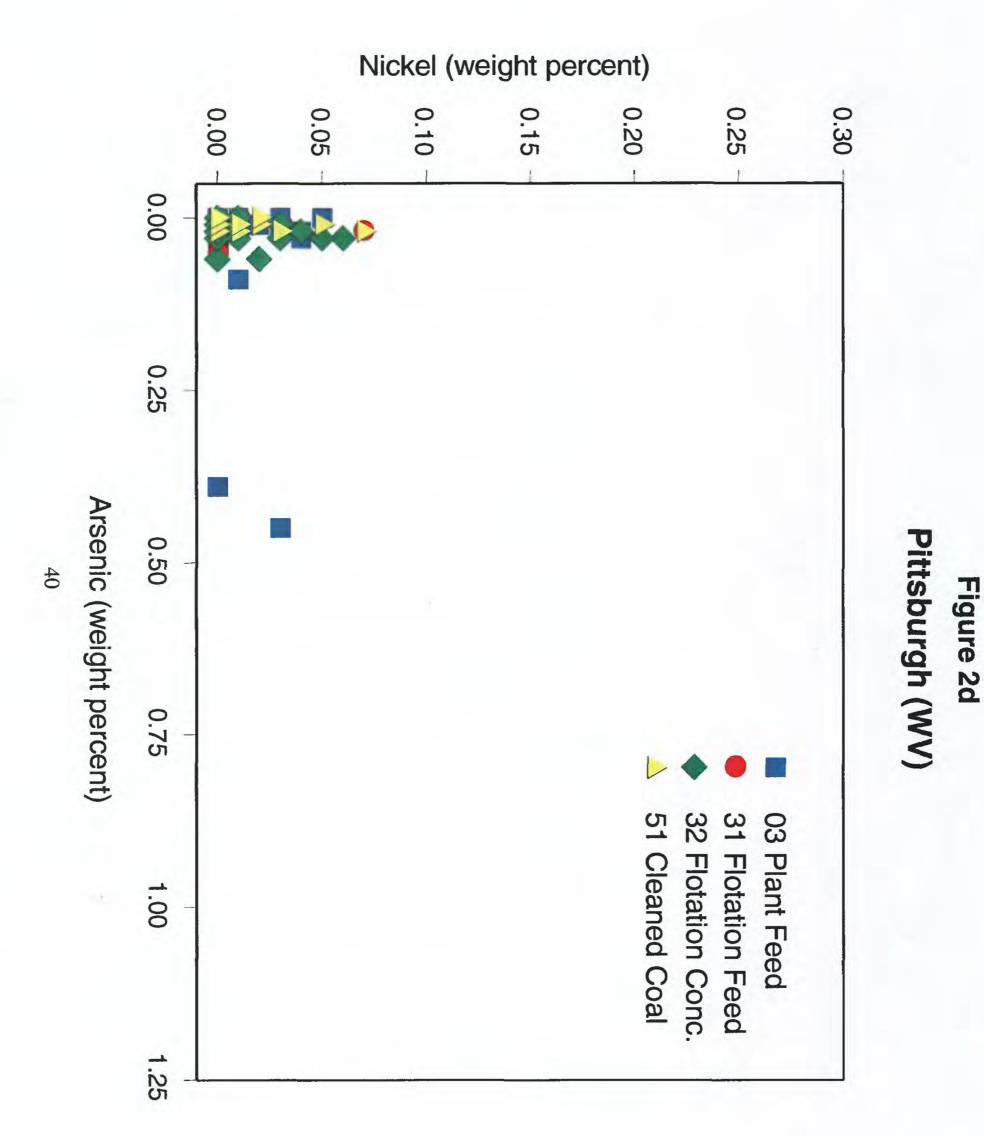


Figure 2c



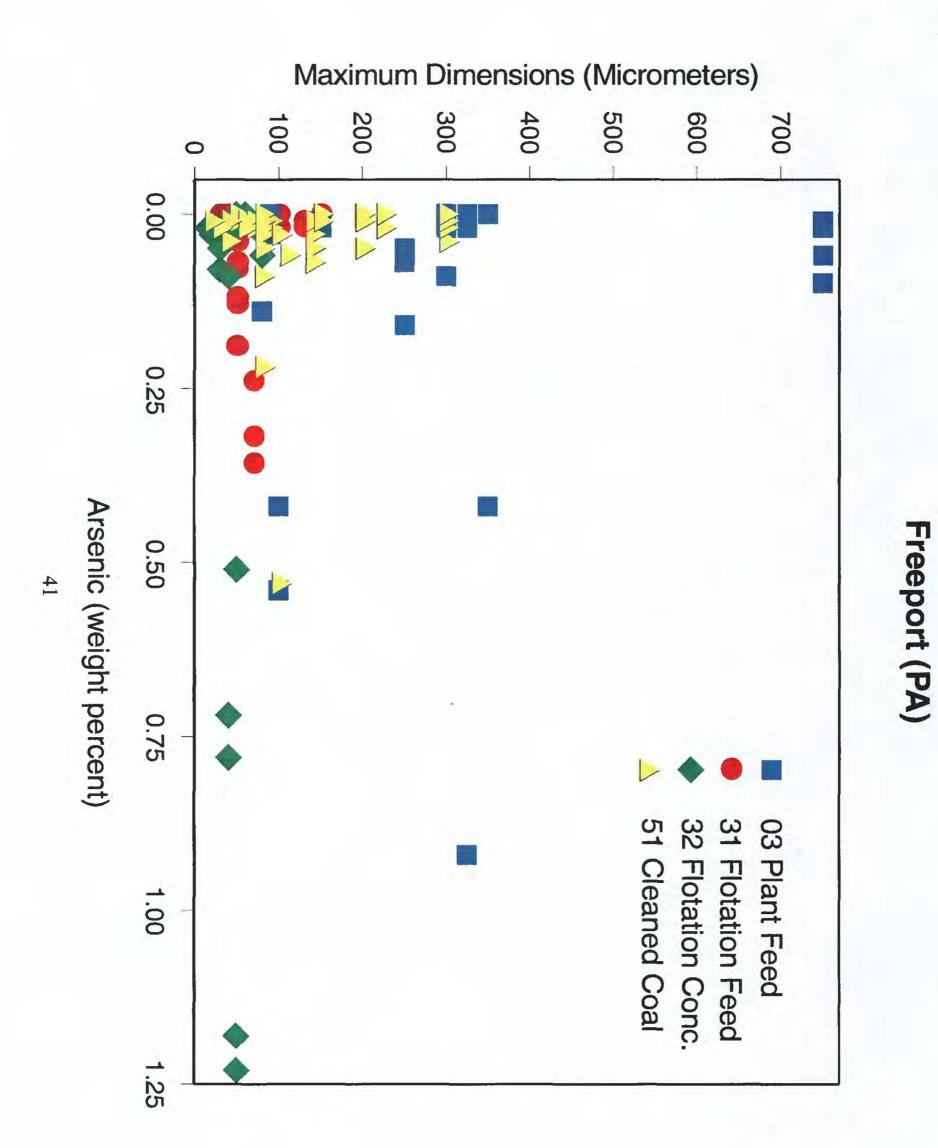


Figure 3a

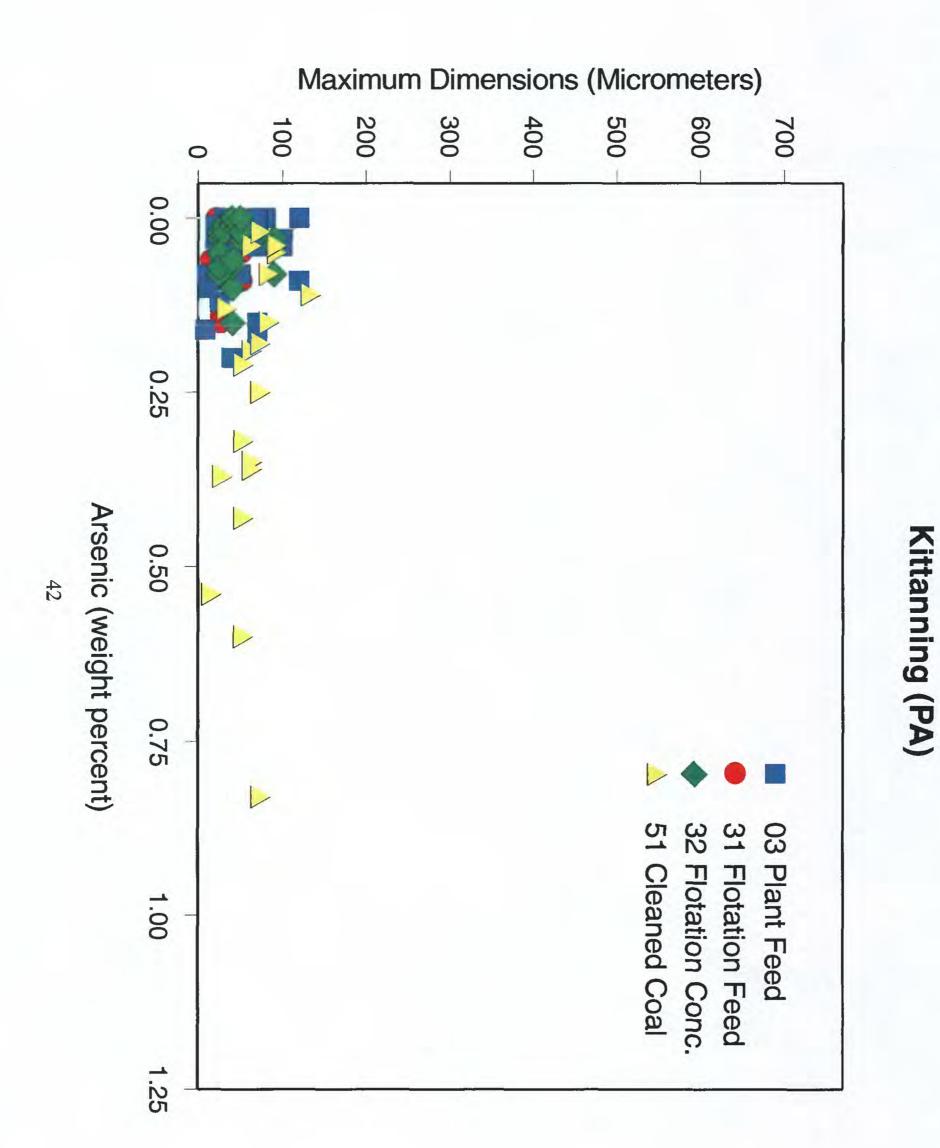


Figure 3b

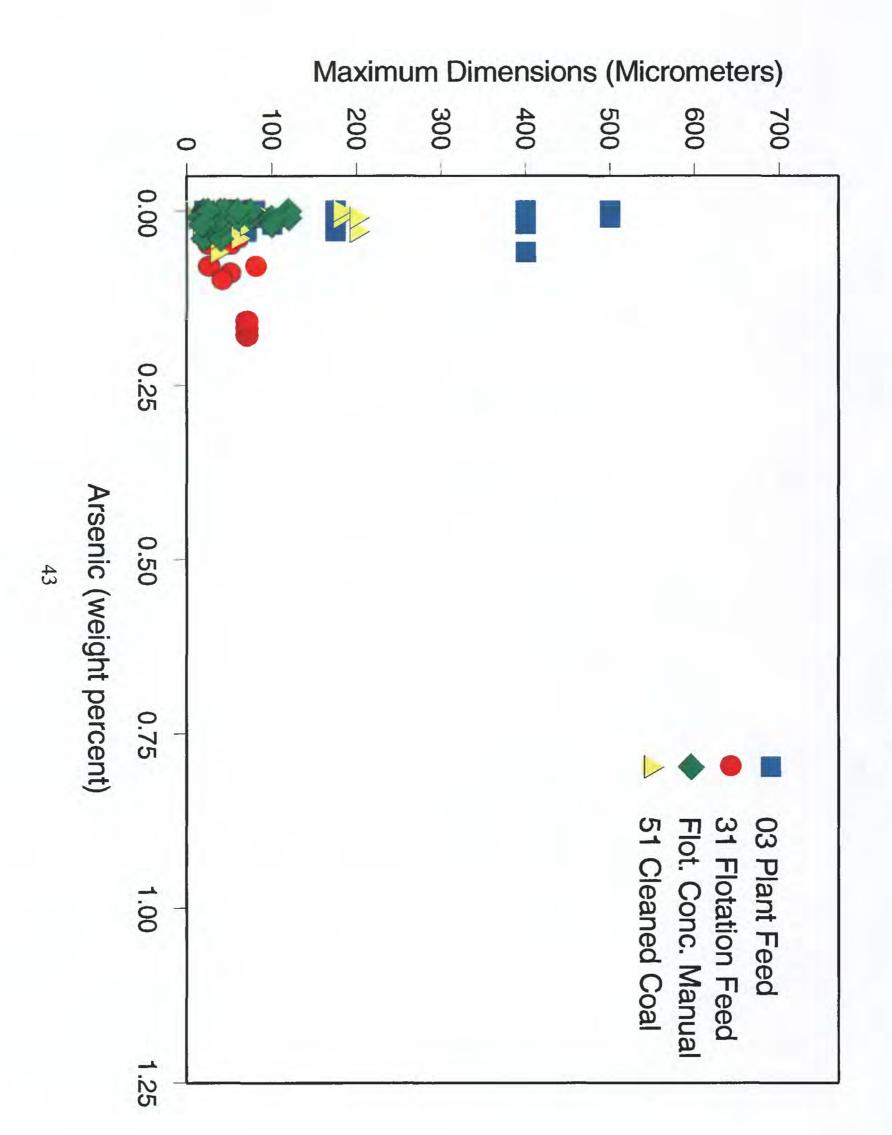


Figure 3c
Pittsburgh (PA)

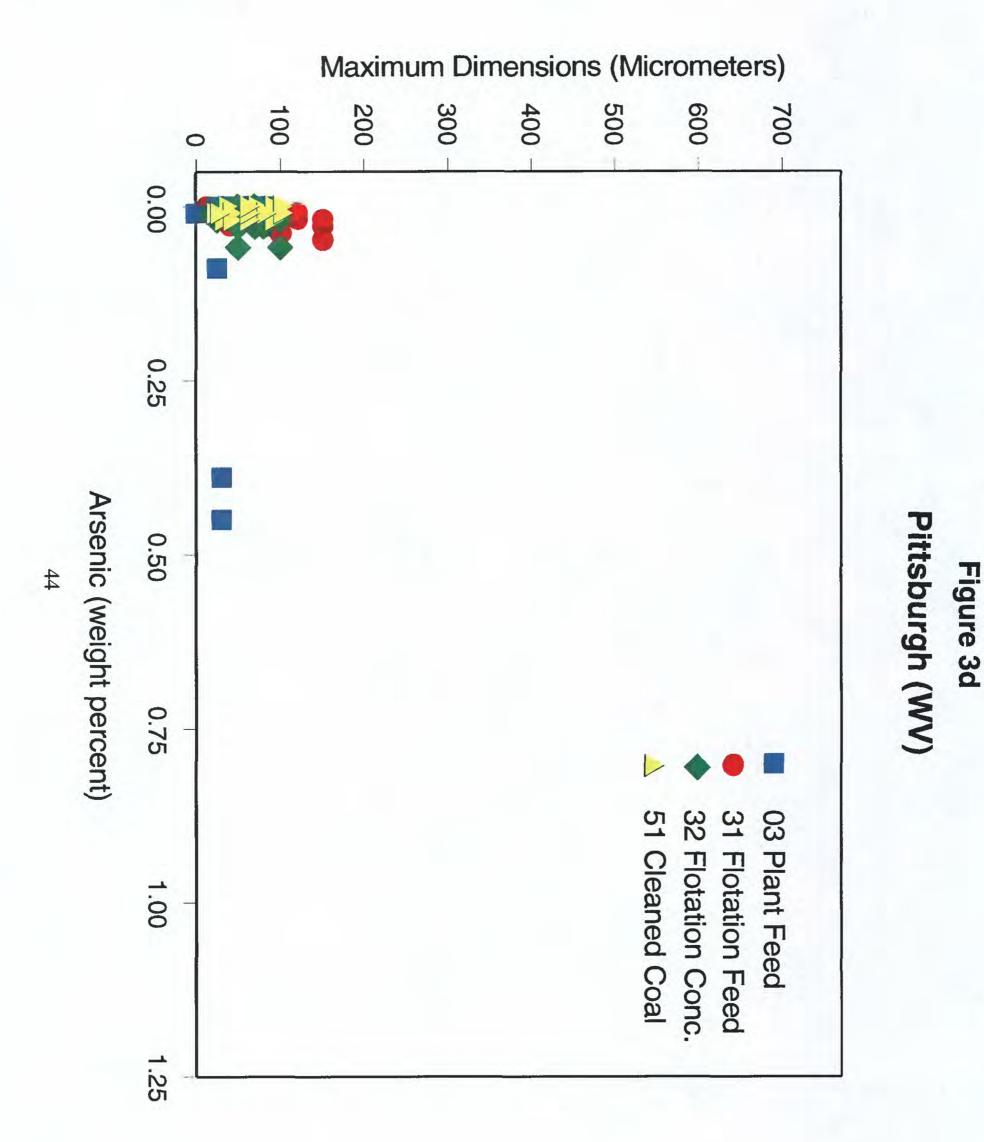


Figure 4

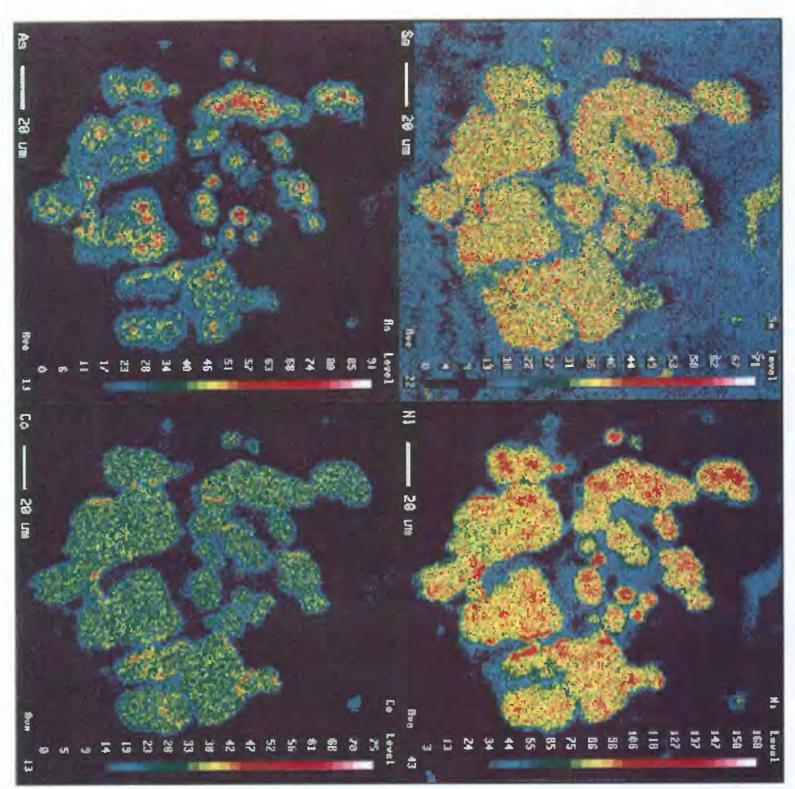


Figure 5

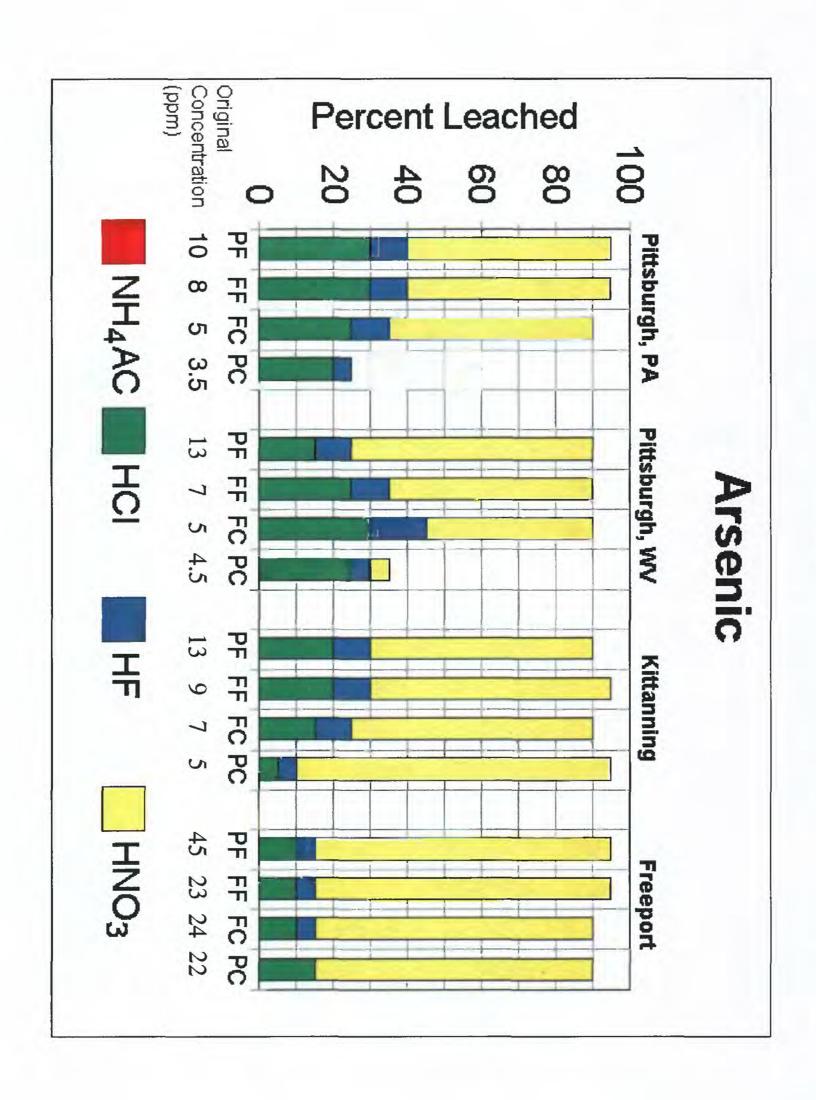


Figure 6

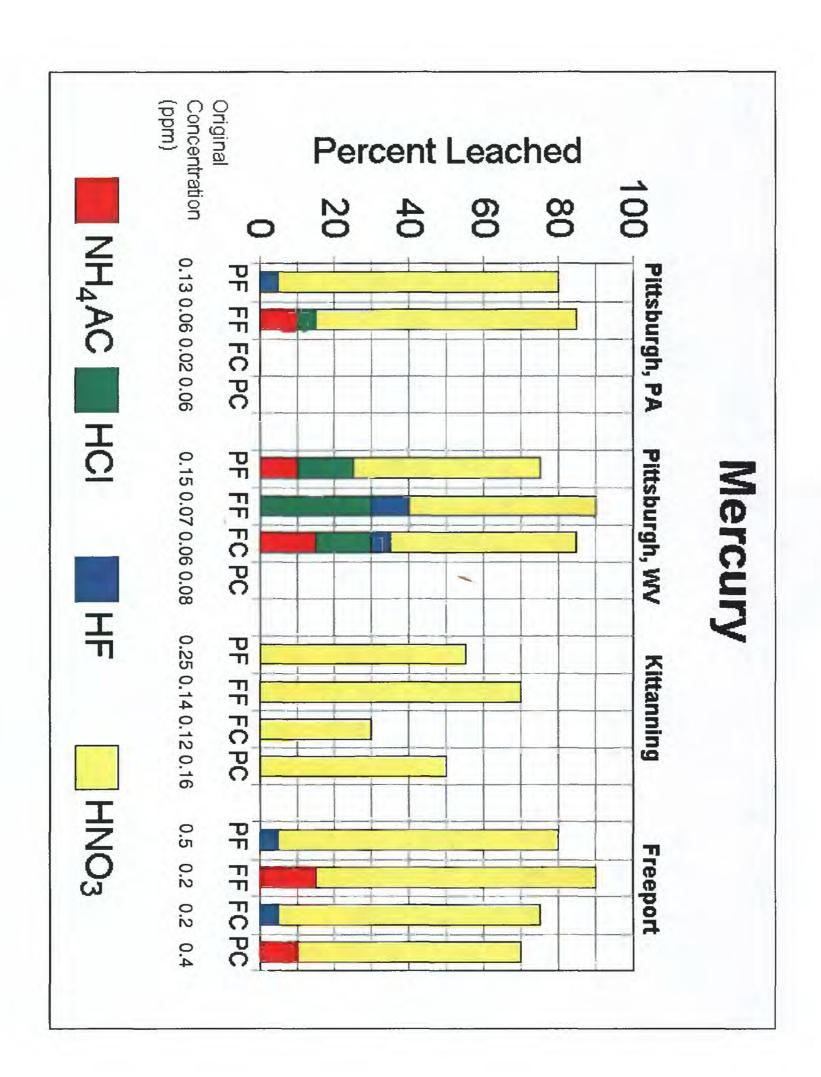
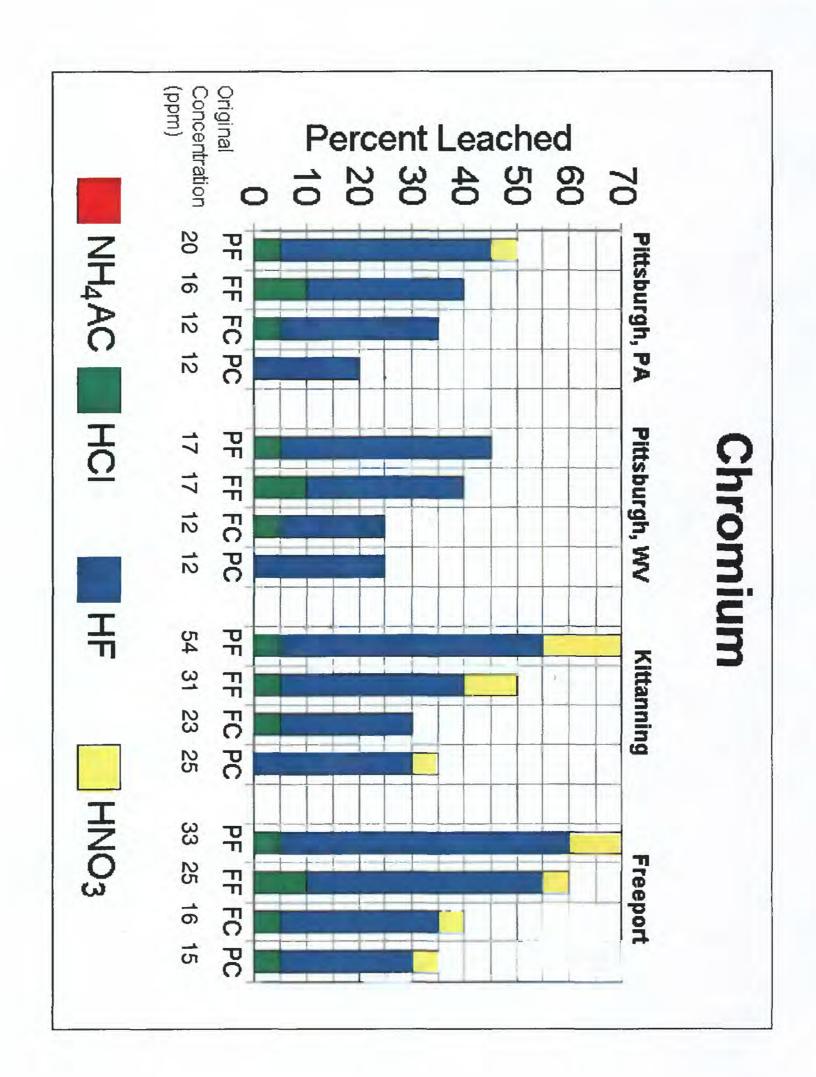


Figure 7



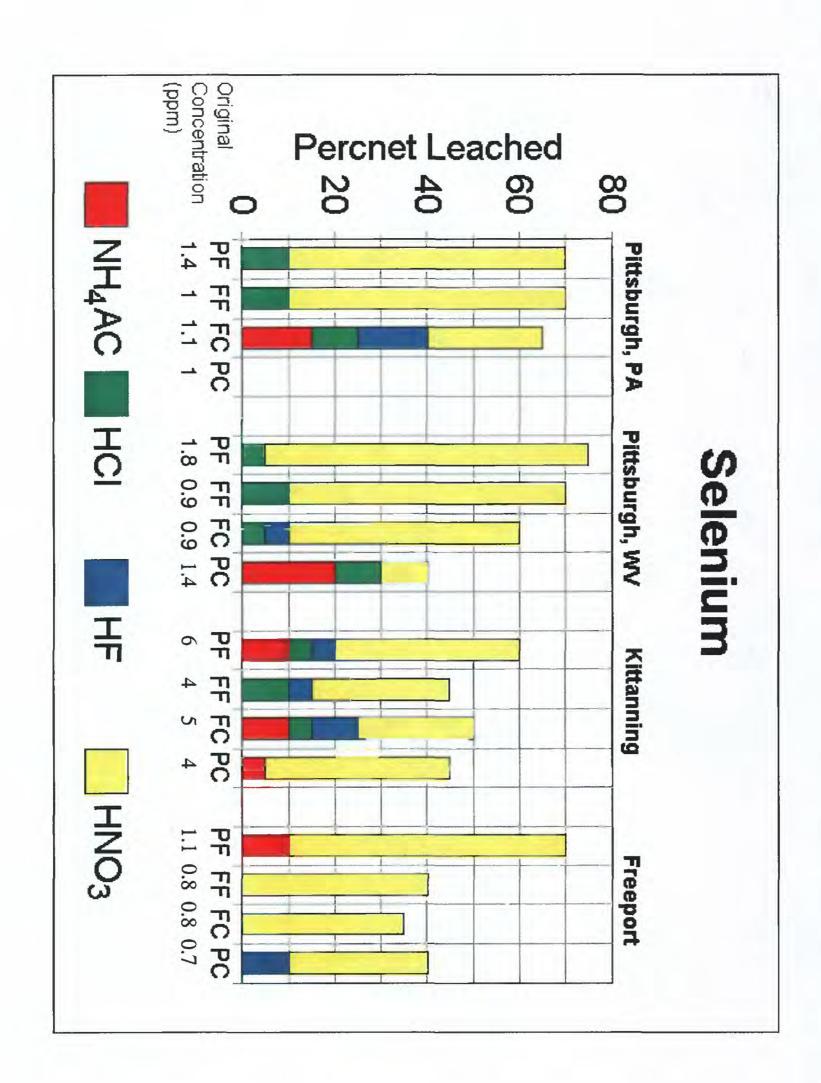


Figure 9

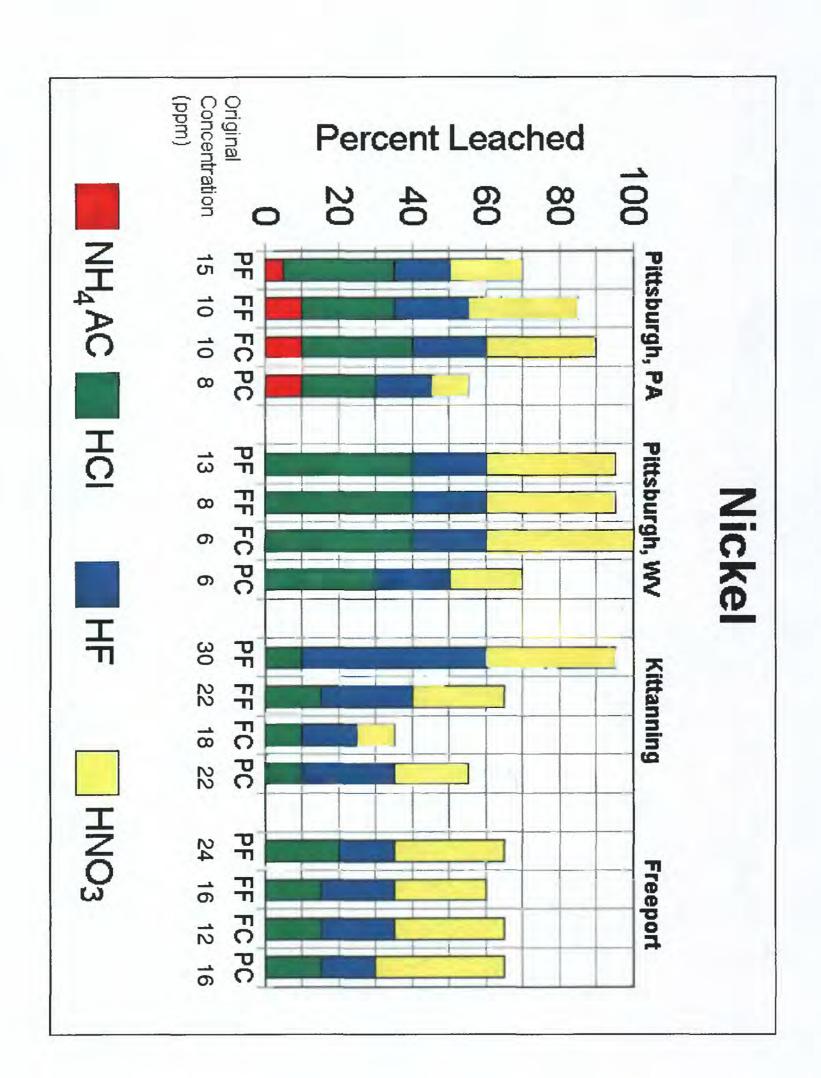


Figure 10

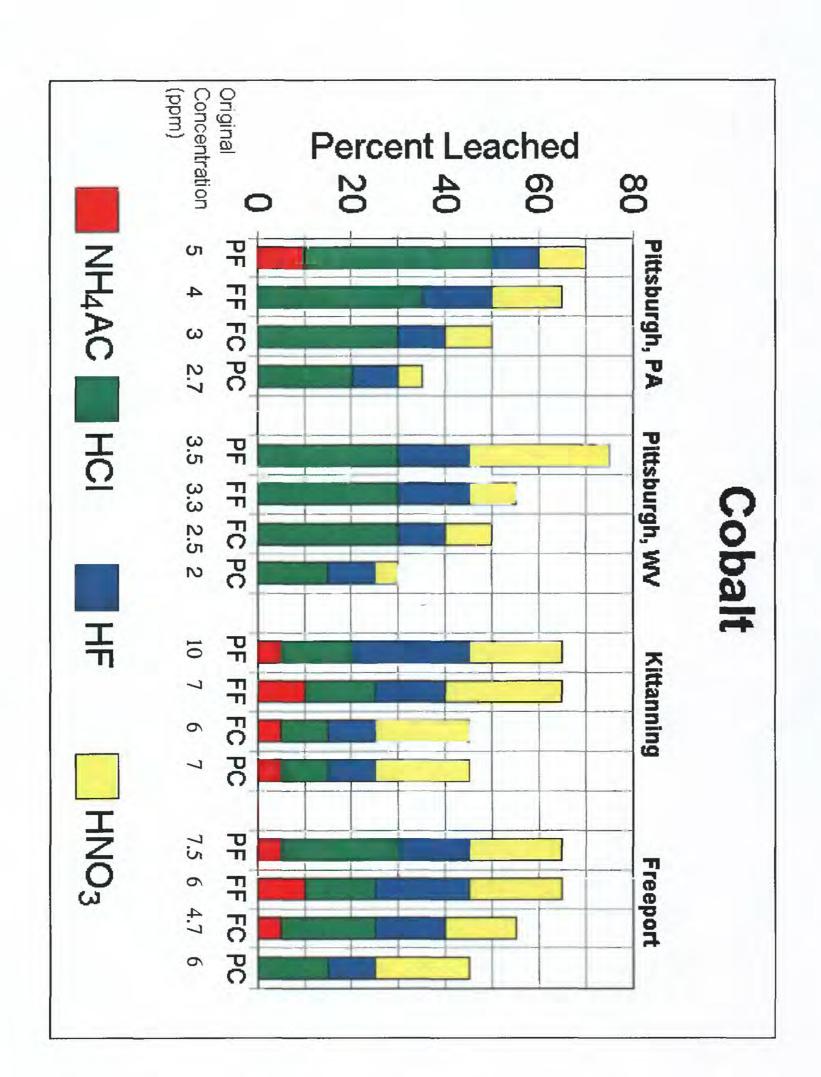


Figure 11

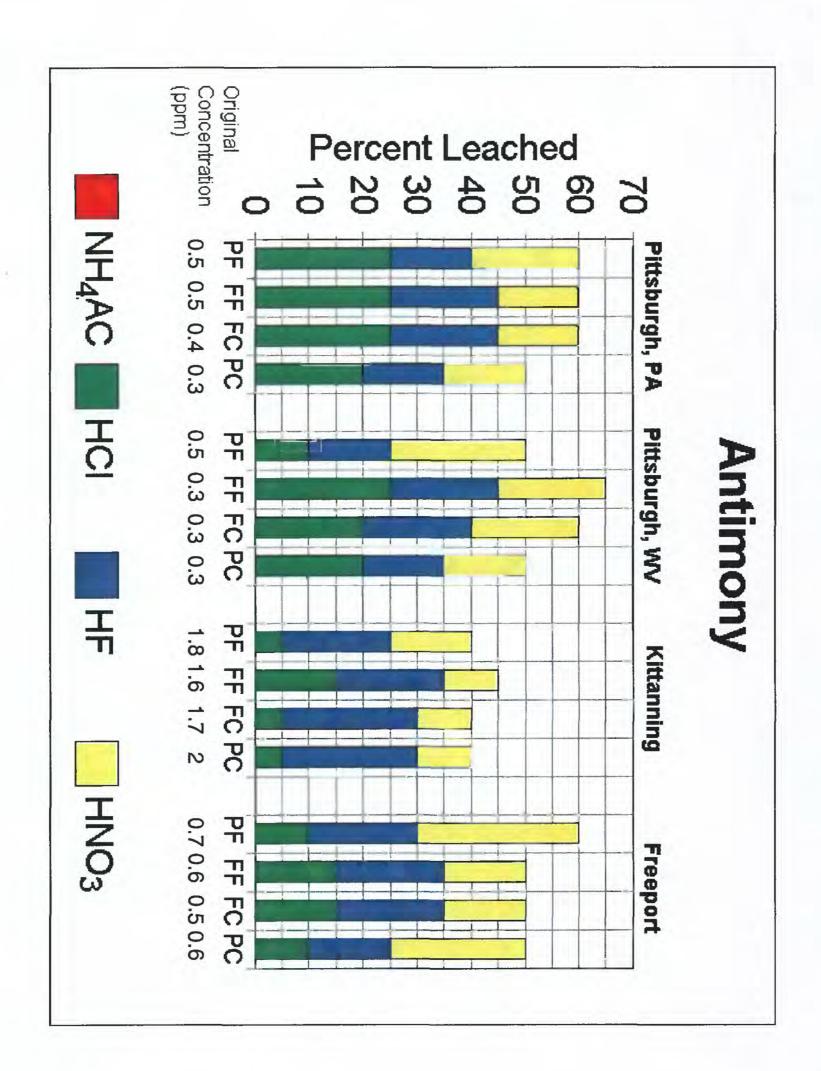


Figure 12

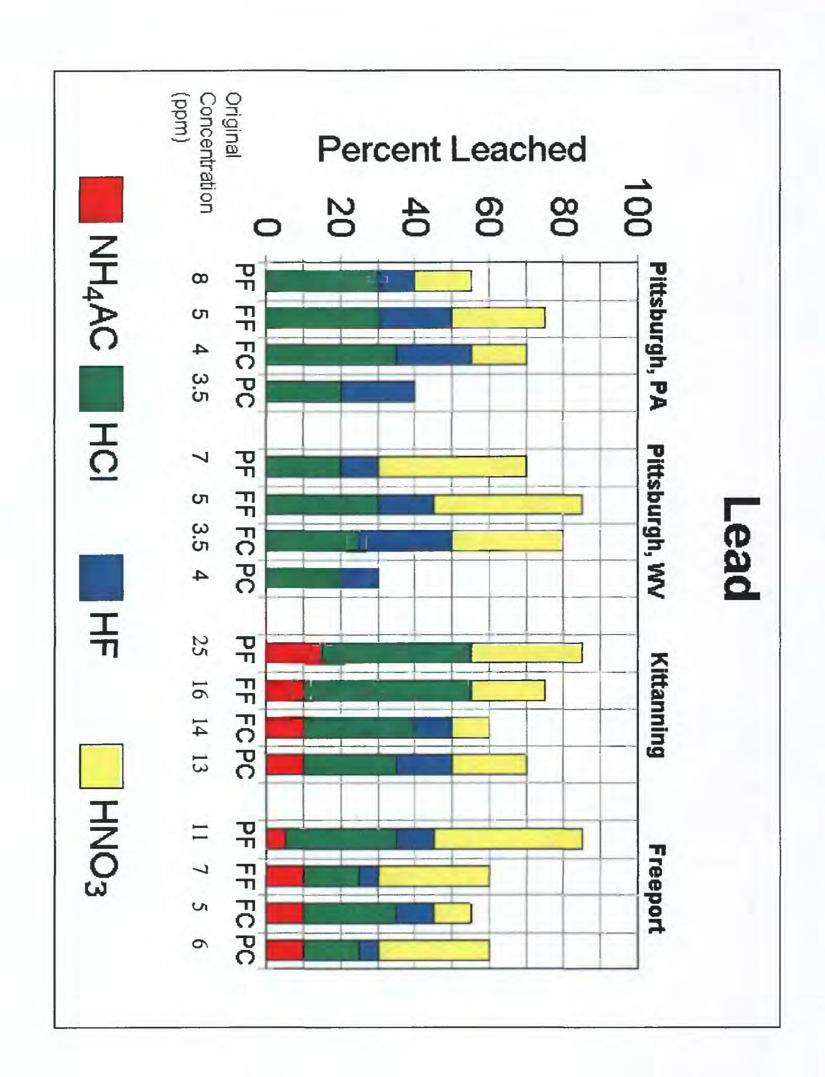


Figure 13

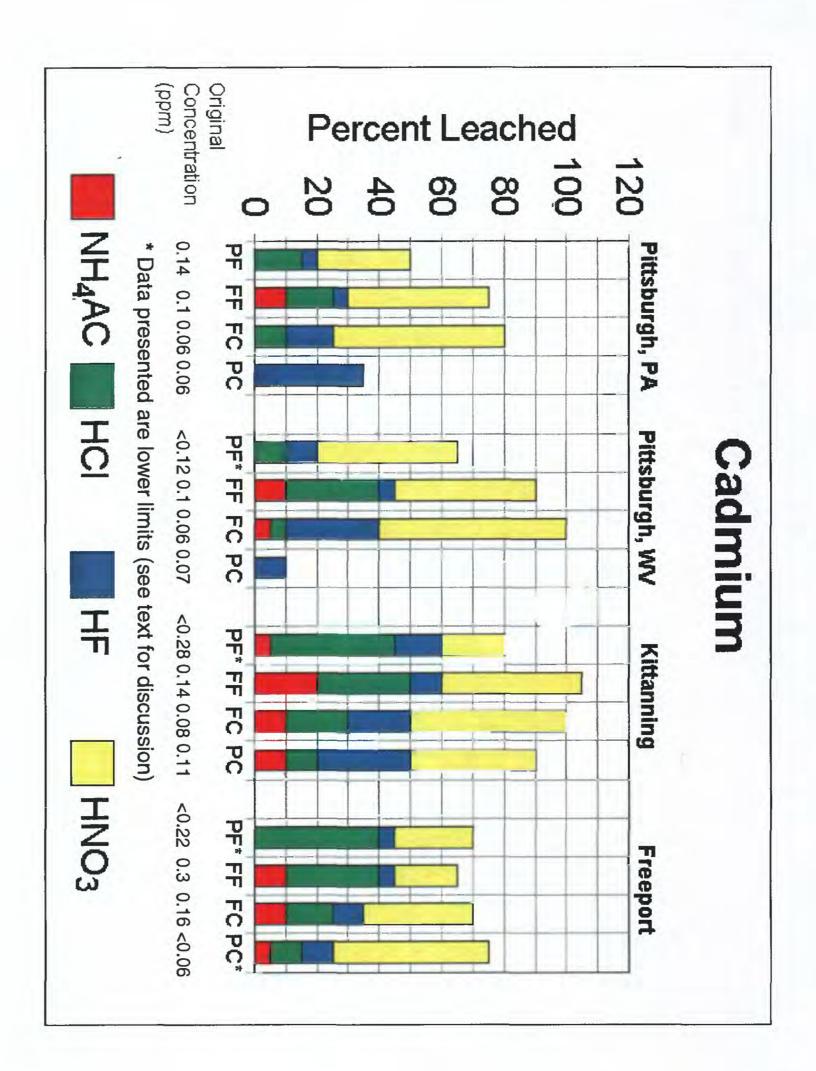


Figure 14

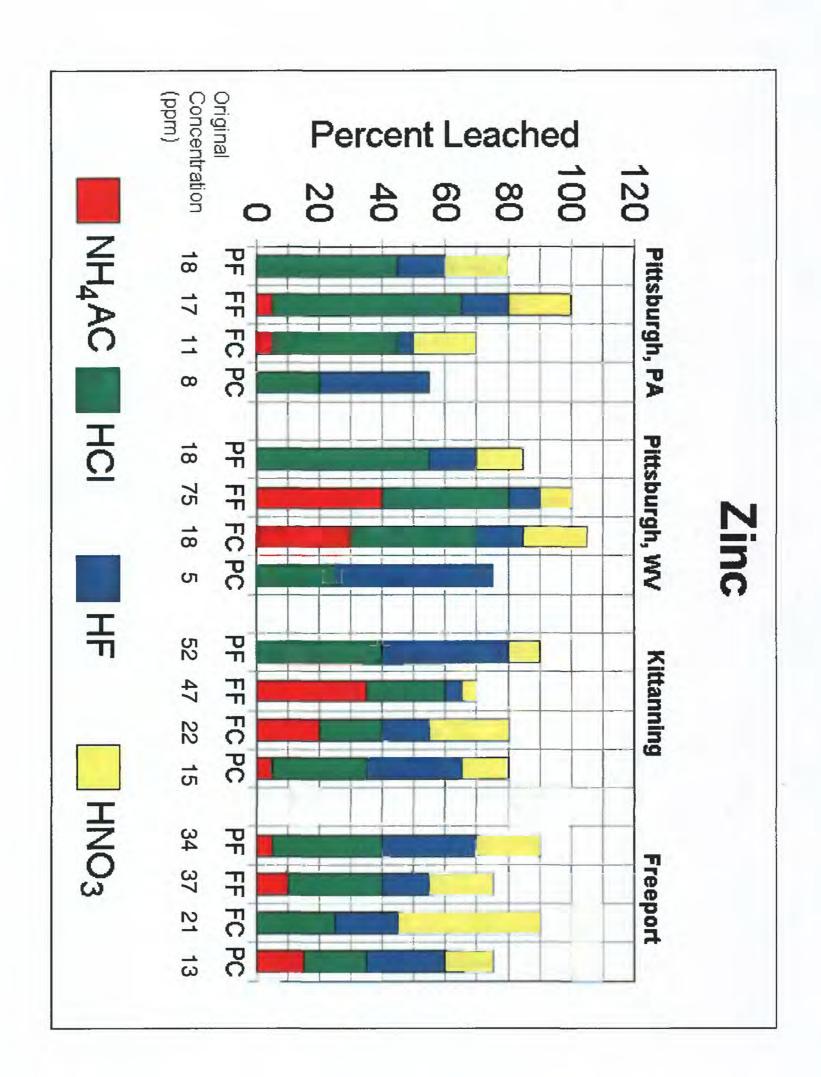


Figure 15

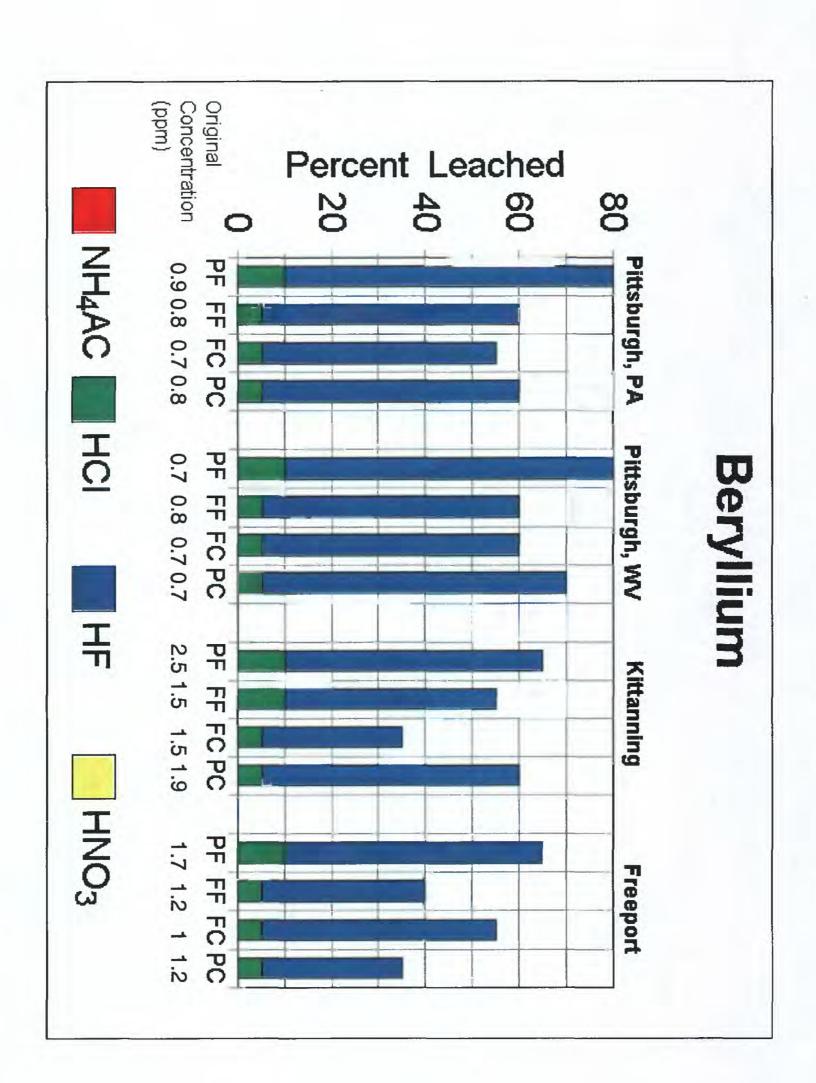


Figure 16

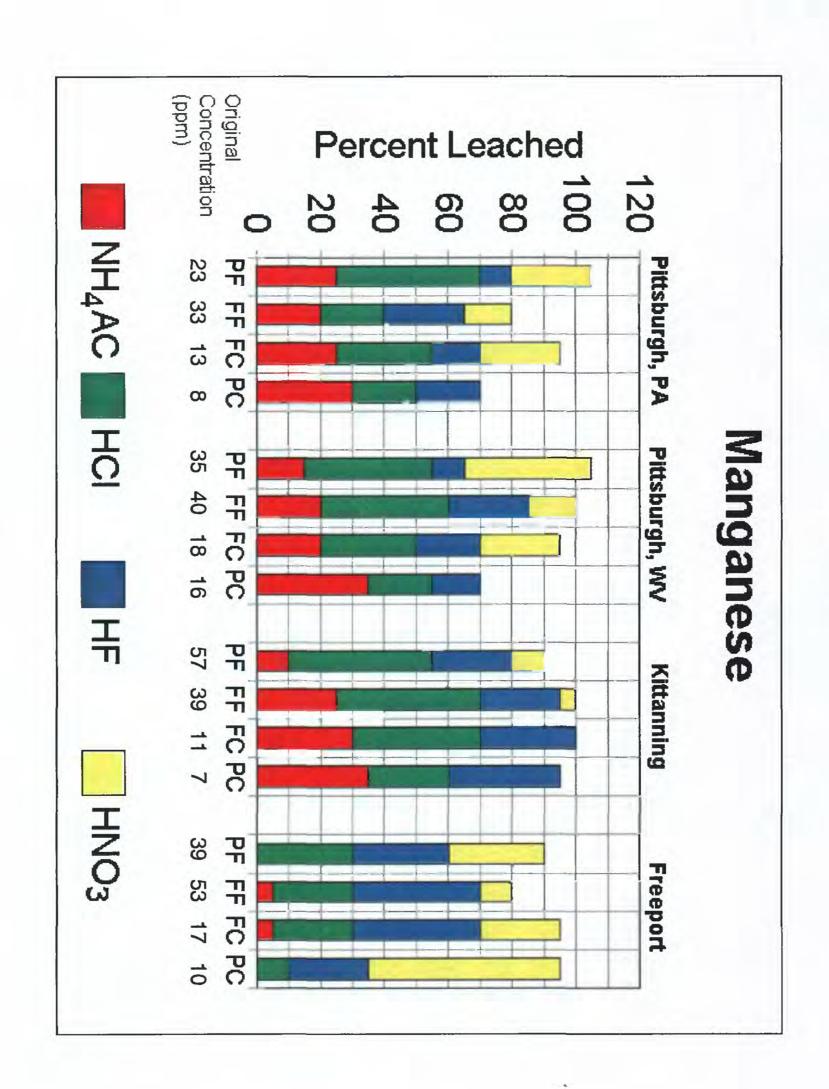


Figure 17

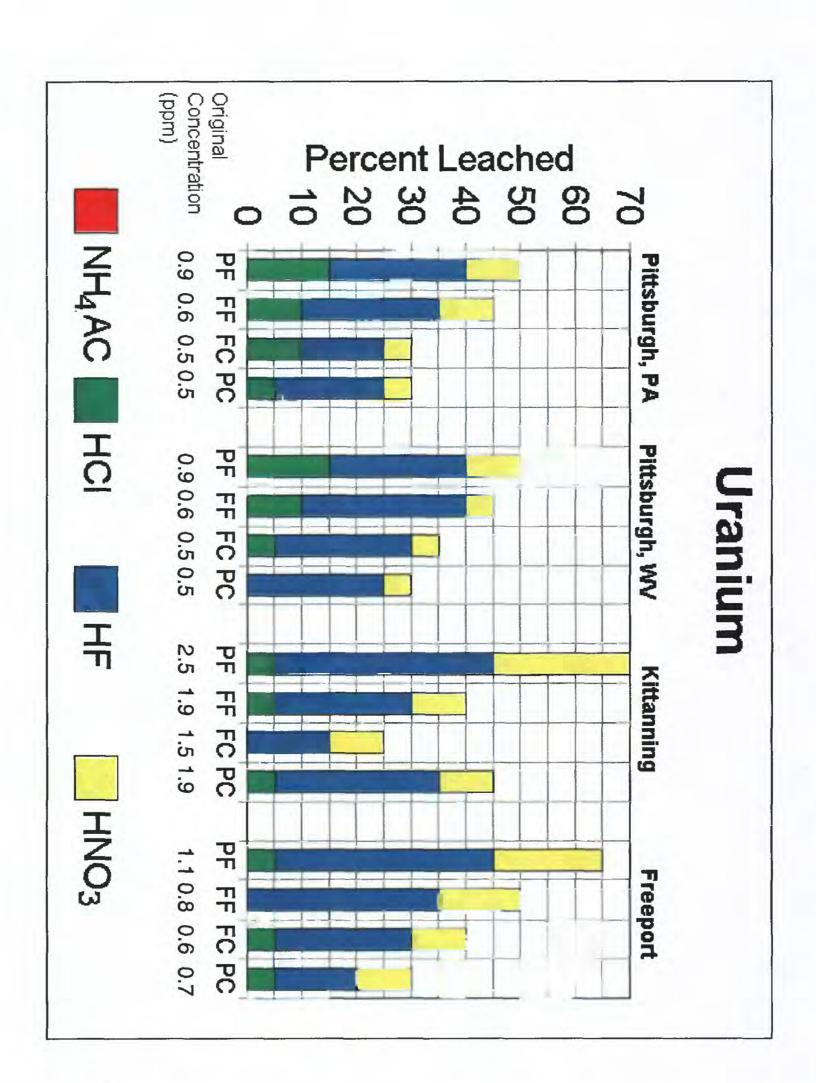


Figure 18

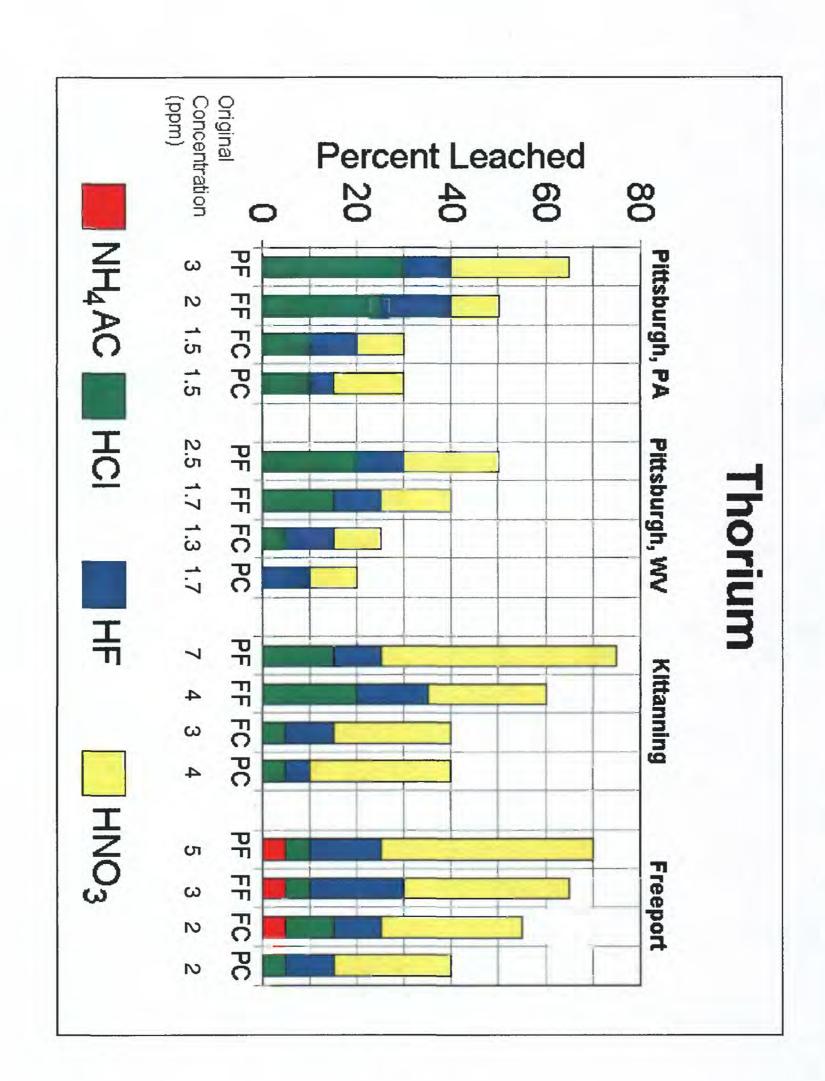
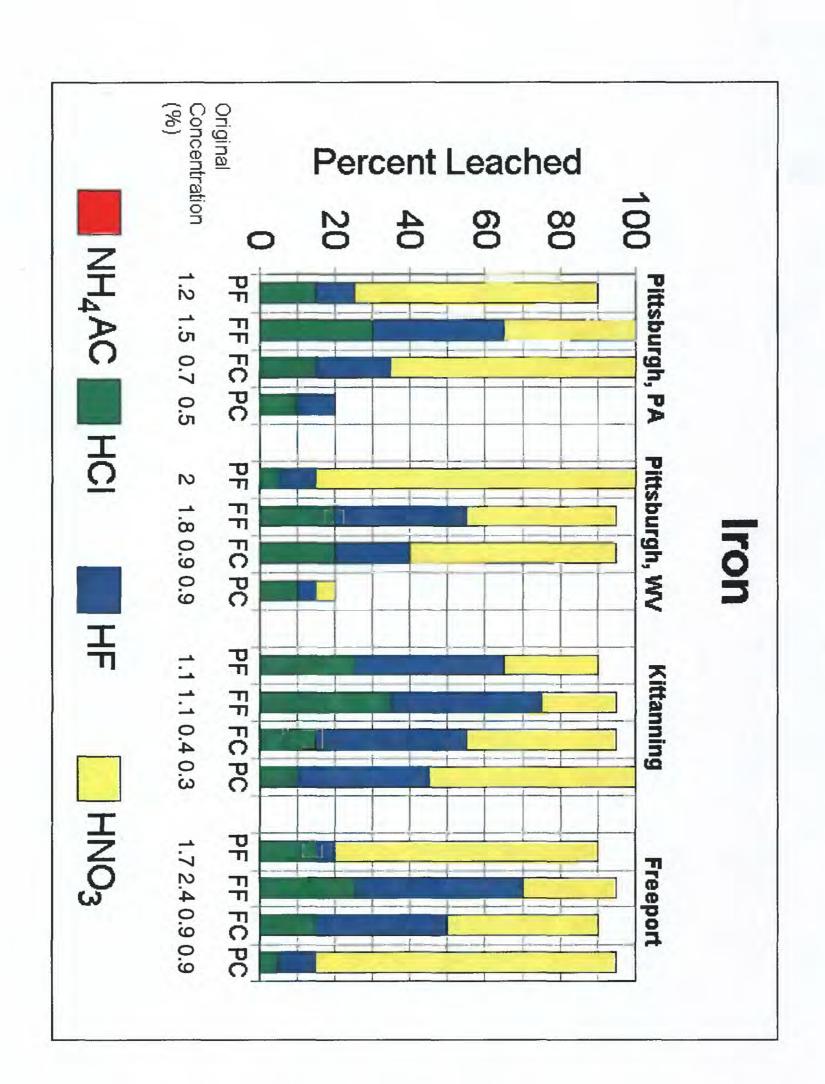


Figure 19



Appendix I

Mineralogy of Feed Coals and Intermediates Based on SEM Analysis

96052401 Pittsburgh Coal (PA)

Major Phases

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Illite	Illite	Illite	Illite
Kaolinite	Kaolinite	Kaolinite	Kaolinite
Quartz	Quartz	Quartz	Quartz
Calcite	Calcite	Calcite	Calcite
Pyrite	Pyrite	Pyrite	Pyrite
	Fe-oxide		

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Chalcopyrite	Chalcopyrite	Fe-oxide	Chalcopyrite
TiO ₂	TiO ₂	TiO ₂	TiO ₂
Barite	Barite	Barite	Fe-oxide
Zircon	Zircon	Zircon	Zircon
Xenotime (Y(PO ₄))	Xenotime	Sphalerite	Sphalerite
Monazite			Galena

96032701 Pittsburgh Coal (WV)

Major Phases

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Illite	Illite	Illite	Illite
Kaolinite	Kaolinite	Kaolinite	Kaolinite
Quartz	Quartz	Quartz	Quartz
Calcite	Calcite	Calcite	
Pyrite	Pyrite	Pyrite	Pyrite

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Fe oxide	Fe oxide	Fe oxide	
Chalcopyrite	Zircon		Zircon
TiO ₂	Sphalerite		TiO ₂
Sphalerite	Sylvite (KCI)		Apatite

96032801 Kittanning Coal Zone(PA)

Major Phases

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Illite	Illite	Illite	Illite
Kaolinite	Kaolinite	Kaolinite	Kaolinite
Quartz	Quartz	Quartz	Quartz
Calcite	Calcite		
Pyrite	Pyrite	Pyrite	Pyrite
	Fe-oxide		

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Chalcopyrite	Chalcopyrite	Chalcopyrite	Chalcopyrite
TiO ₂		TiO ₂	TiO ₂
Zircon			Zircon
Monazite	Monazite		Monazite
Barite	Barite	Fe-oxide	Fe-oxide
Sphalerite		Sphalerite	Xenotime
Galena		Galena	Pb selenide

96032901 Freeport Coal Zone(PA)

Major Phases

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Illite	Illite	Illite	Illite
Kaolinite	Kaolinite	Kaolinite	Kaolinite
Quartz	Quartz	Quartz	Quartz
Pyrite	Pyrite	Pyrite	Pyrite
Fe-oxide	Fe-oxide	Fe-oxide	

Plant Feed	Froth. Feed	Froth. Conc.	Plant Clean
Chalcopyrite			Fe-oxide
TiO ₂	TiO ₂	TiO ₂	TiO ₂
Zircon		Zircon	Zircon
	Monazite	Monazite	Monazite
	Epidote (?)	Sphalerite	Sphalerite
Galena			Apatite

Appendix II Quantitative Microprobe Analyses

A. Pyrite Analyses

FREEPORT Coal Zone (PA)

	37	36	35	34	ၓၟ	32	<u> </u>	30	29	28	27	26	25	24	23	22	21	20	19	15	4	3	12	11	10	9		No.
	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01**		Se
	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.01	0.00	0.01	0.02	0.02	0.00	0.01	0.00	0.00	0.00		δ
	0.00	0.00	0.01	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.02	0.02	0.02	0.00	0.01	0.00		<u>z</u>
	0.01	0.01	0.04	0.02	0.01	0.00	0.00	0.02	0.00	0.07	0.08	0.00	0.00	0.02	0.19	0.02	0.01	0.01	0.00	0.00	0.12	0.13	0.08	0.03	0.01	0.00		As
<u> </u>	0.00	0.01	0.02	0.02	0.03	0.01	0.00	0.00	0.01	0.00	0.02	0.02	0.02	0.00	0.01	0.03	0.02	0.02	0.00	0.03	0.00	0.00	0.00	0.00	0.02	0.01		Zn
FREEPORT	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	31 FI	S
	0.04	0.05	0.05	0.04	0.05	0.05	0.04	0.05	0.04	0.04	0.04	0.05	0.05	0.05	0.05	0.04	0.06	0.06	0.05	0.06	0.05	0.06	0.06	0.06	0.05	0.05	Flotation Feed	٥ ک
																											$\overline{\mathcal{L}}$	
Coal Zone (PA)	46.00	46.28	46.24	45.91	47.88	47.57	46.60	46.80	47.21	47.06	47.17	47.04	47.33	47.77	46.53	46.95	47.01	47.25	47.26	46.48	46.39	46.60	46.82	47.61	47.54	47.80	Feed	Fe
one (PA)						47.57 52.22																					Feed	Fe S
one (PA)	51.58	51.53	50.53	51.48	52.48		51.02	51.35	50.77	51.62	51.96	52.39	52.63	52.32	50.82	52.25	52.18	52.03	52.74	51.40	51.32	51.57	51.92	51.84	52.06		Feed	
one (PA)	51.58	51.53	50.53	51.48	52.48	52.22	51.02	51.35 98.23	50.77	51.62 98.79	51.96	52.39	52.63	52.32 100.18	50.82 97.61	52.25	52.18 99.29	52.03	52.74 100.06	51.40 98.00	51.32 97.92	51.57	51.92 98.91	51.84	52.06 99.69	52.15	Feed	တ

	65 5																48		44					39		No.
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.01	Se
0.01	0.05	0.01	0.02	0.02	0.00	0.00	0.00	0.00	0.04	0.00	0.02	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	င်
0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.26	0.00	0.00	0.04	0.00	0.00	0.01	0.00	0.02	0.00	0.02	0.01		0.00	0.01	0.00	0.00	Z
0.05	0.08	0.02	0.01	0.01	0.92	0.01	0.00	0.02	0.10	0.01	0.02	0.06	0.01	0.00	0.09	0.02	0.00	0.00	0.42	0.00		0.36	0.32	0.24	0.00	As
0.01	0.01	0.01	0.02	0.02	0.02	0.02	0.00	0.01	0.04	0.02	0.01	0.02	0.01	0.02	0.01	0.03	0.03	0.01	0.01	0.02		0.02	0.01	0.01	0.00	Zn
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	03 F	0.00	0.00	0.00	0.00	Ω
0.03	0 0	0.0	0.	0.	0.	0.0	0.0	0.0	0.0	0.	0	0	0	0	0	0	0	0.05	0	0	lant	0	0	0.04	0	ဂ္ပ
	0 4	4	04	2	2	4	5	4	55	05	2	95	2	2	4	05	8	05	2	2	Fee	2	94	4	4	o _*
46.32	4 46.50																		•	04 46.77	03 Plant Feed			0 4 47.57		o * Fe
		47.14	47.34	47.51	47.03	46.80	46.73	47.15	45.20	46.75	46.35	46.08	47.10	46.22	46.40	46.29	46.80	46.90	44.21		Feed	47.31	46.87		46.40	7
50.74	46.50 46.84	47.14 52.79	47.34 52.83	47.51 52.19	47.03 51.30	46.80 51.46	46.73 51.89	47.15 51.71	45.20 49.79	46.75 50.96	46.35 51.14	46.08 51.47	47.10 51.92	46.22 50.76	46.40 51.34	46.29 50.50	46.80 51.20	46.90 50.94	44.21 49.61	46.77	Feed	47.31 52.29	46.87 51.54	47.57	46.40 51.64	т e
50.74 97.17	46.84 51.95 98.96	47.14 52.79 100.00	47.34 52.83 100.25	47.51 52.19 99.79	47.03 51.30 99.31	46.80 51.46 98.34	46.73 51.89	47.15 51.71 98.94	45.20 49.79 95.49	46.75 50.96 97.80	46.35 51.14 97.57	46.08 51.47 97.73	47.10 51.92 99.07	46.22 50.76 97.04	46.40 51.34 97.90	46.29 50.50 96.89	46.80 51.20	46.90 50.94 97.91	44.21 49.61 94.31	46.77 50.88	Feed	47.31 52.29 100.02	46.87 51.54	47.57 52.39 100.24	46.40 51.64	Fe S

FREEPORT Coal Zone (PA)

	89 90 91 92	86 87	8 8 4 4 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	78 79	75 76 77	70 71	67	N 0
0.01 0.00 0.00 0.00	0.00	0.00	0.00	0.00	0.00 0.01 0.00	0.00	0.00	Se
0.00 0.01 0.00 0.00	0.01 0.00 0.00 0.02	0.00 0.12 0.18	0.01 0.00 0.01	0.00	0.10 0.02 0.06	0.00	0.00	ပ
0.00 0.01 0.00 0.00	0.00 0.00 0.00	0.01 0.14 0.01	0.00 0.00 0.05	0.00	0.02 0.00 0.00	0.01	0.00	<u>z</u>
0.02 0.01 1.23 1.18 0.51	0.02 0.02 0.00	0.00	0.02 0.03 0.02	0.08	0.00 0.02 0.03	0.14	0.16	As
0.00 0.00 0.03 0.04 0.02	0.01 0.05 0.03	0.01	0.01 0.00 0.02 0.02	0.02	0.02 0.02 0.00	0.01	0.02	Zn
0.00 0.00 0.00	0.00 0.00 0.00	0.00	0.00 0.00	32 Flotation Concentrate 0.00	0.00 0.00	0.00	0.00	Cd
0.04 0.04 0.04 0.05 0.03	0.04 0.04 0.04	0.04	0.05 0.04 0.07 0.04	0.05	0.04 0.04	0.04	0.05	င္ပ
				Q				
47.16 46.01 47.41 47.25 47.25	47.09 47.52 47.27 46.66	47.17 44.29 46.78	46.89 47.24 44.95 47.35	centrate 47.01 45.78	46.44 46.67 47.10	47.33 46.56 47.29	46.99 47.56	Fe
47.1651.9646.0150.4247.4151.0147.2552.3147.2252.37			46.89 50.92 47.24 52.10 44.95 49.98 47.35 51.46	& 4	46.44 50.84 46.67 51.08 47.10 51.80			Fe S
	51.58 98.75 51.91 99.53 51.29 98.64 51.81 98.63	51.43 49.91 51.27		1 51.69 8 49.06		51.82 52.45	52.16 52.23	
51.96 99.21 50.42 96.52 51.01 99.72 52.31 100.82 52.37 100.15	51.58 98.75 51.91 99.53 51.29 98.64 51.81 98.63	51.43 98.66 49.91 94.60 51.27 98.33	50.92 97.89 52.10 99.42 49.98 95.10 51.46 98.88	1 51.69 98.86 8 49.06 94.99	50.84 97.46 51.08 97.86 51.80 99.04	51.82 52.45	52.16 99.40 52.23 100.26	တ

FREEPORT Coal Zone (PA)

94	93	92	91	90	89	88	87	86	85	84	83	82	81	80	79	78	77	76	75	73		102	101	100	99	98	N _o
0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.01	0.00	0.00		0.00	0.01	0.00	0.00	0.00	Se
0.07	0.08	0.00	0.04	0.04	0.04	0.02	0.00	0.00	0.01	0.01	0.00	0.00	0.01	0.00	0.02	0.02	0.00	0.01	0.00	0.01		0.00	0.00	0.00	0.00	0.00	င္
0.02	0.01	0.02	0.00	0.02	0.06	0.00	0.02	0.01	0.01	0.02	0.00	0.01	0.02	0.00	0.00	0.01	0.05	0.01	0.01	0.02		0.02	0.01	0.01	0.00	0.00	<u>z</u>
0.04	0.02	0.01	0.01	0.00	0.04	0.01	0.06	0.06	0.02	0.01	0.01	0.02	0.06	0.05	0.03	0.00	0.05	0.01	0.00	0.02		0.01	0.02	0.02	0.72	0.78	As
0.03	0.03	0.04	0.00	0.02	0.00	0.02	0.03	0.01	0.01	0.01	0.01	0.00	0.00	0.00	0.01	0.00	0.02	0.02	0.02	0.01		0.02	0.03	0.01	0.01	0.02	Zn
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	51 C	0.00	0.00	0.00	0.00	0.00	C
0.04	0.0	0.05	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	eane	0.0	0.04	0.0	0.0	0.0	င့
_	Gi	GI	Oi	0	တ	(J	4	4	G	G	6	တ	()	Ö	ŰΊ	Ö	Ö	4	Ö	<u></u>	O O	4	4	4	4	4	*
47.33							4 47.47											•			Cleaned Coal		•	4 46.97	•)* Пе
_	47.66	48.21	47.39	47.60	46.44	47.80		47.29	47.32	46.74	47.91	47.22	46.96	47.42	47.47	47.28	47.71	47.66	47.29	44.99	d Coal	45.43	45.77	•	47.68	47.77	-
47.33	47.66 51.58	48.21 52.00	47.39 51.94	47.60 51.43	46.44 50.89	47.80 51.69	47.47	47.29 51.78	47.32 50.84	46.74 50.58	47.91 51.61	47.22 50.77	46.96 51.06	47.42 51.79	47.47 51.79	47.28 50.92	47.71 51.42	47.66 51.16	47.29 51.02	44.99 50.53	d Coal	45.43 49.52	45.77 49.77	46.97	47.68 51.66	47.77 52.04	Fe
47.33 51.42	47.66 51.58 99.42	48.21 52.00	47.39 51.94	47.60 51.43	46.44 50.89 97.54	47.80 51.69	47.47 51.42	47.29 51.78 99.19	47.32 50.84 98.26	46.74 50.58	47.91 51.61	47.22 50.77	46.96 51.06	47.42 51.79	47.47 51.79	47.28 50.92 98.28	47.71 51.42 99.31	47.66 51.16	47.29 51.02 98.40	44.99 50.53	d Coal	45.43 49.52	45.77 49.77	46.97 51.19	47.68 51.66 100.12	47.77 52.04 100.64	Fe

No.	Se	ပ်	<u>Z</u>	As	Zn	S	°,	Fe	တ	Total	Grain	Size(microns)/form
95	0.01					0.00	0.06	47.38	51.14	98.68	Py6.4	40 framboid
96	0.00					0.00	0.05	46.95	51.21	98.39	Py6.5	40 framboid
102	0.00	0.01				0.00	0.06	47.29	51.91	99.28	Py9.1	60 x 80 euhedral
103	0.01					0.00	0.05	47.75	52.29	100.18	Py9.2	
104	0.00					0.00	0.05	47.19	51.77	99.10	Py9.3	
106	0.01					0.00	0.05	47.49	51.48	99.07	Py10.2	70x140 irregular
108	0.00					0.00	0.05	47.57	51.28	98.95	Py11.1	180x300 subhedral
109	0.00					0.00	0.04	46.99	51.96	99.03	Py11.2	
110	0.00					0.00	0.05	47.14	51.65	98.86	Py11.3	
111	0.00					0.00	0.05	47.90	52.35	100.30	Py11.4	
112	0.00					0.00	0.06	47.38	51.07	98.57	Py12.1	120 x 150 subhedral
113	0.00					0.00	0.04	47.26	51.44	98.79	Py12.2	
114	0.00					0.00	0.05	47.42	51.77	99.27	Py12.3	
115	0.00					0.00	0.05	47.75	52.30	100.10	Py13.1	150 x 225 cleat?
116	0.00					0.00	0.05	47.54	52.00	99.63	Py13.2	
117	0.00					0.00	0.05	47.87	51.94	99.89	Py13.3	
118	0.00					0.00	0.05	47.86	52.31	100.25	Py13.4	
119	0.00					0.00	0.05	47.84	52.22	100.12	Py13.5	
120	0.00					0.00	0.04	46.93	51.10	98.14	Py14.1	70 x 80 subhedral
121	0.00					0.00	0.04	47.33	51.62	99.22	Py14.2	
122	0.00					0.00	0.04	46.88	51.23	98.30	Py14.3	
123	0.00					0.00	0.05	46.98	51.25	98.36	Py15.1	30 euhedral
124	0.00					0.00	0.05	46.71	50.38	97.25	Py16.1	100 composite
125	0.01					0.00	0.05	47.17	50.73	98.08	Py16.2	
126	0.00					0.00	0.11	44.55	48.54	94.06	Py16.3	
127	0.00					0.00	0.05	46.48	50.49	97.19	Py17.1	80 composite
128	0.00					0.00	0.05	46.12	50.08	96.58	Py17.2	
130	0.00		0.00	0.01	0.02	0.00	0.05	47.20	52.43	99.70	Py18.1	20 framboid
Co data i	nclude 0	0.03 to 0.0	_	₽ .	_	nterferend	10					

^{*} Co data include 0.03 to 0.05 wt. % contribution of iron interference.

^{**}Detection limit for other elements is 0.01 wt. %.

	P18.2	98.32	51.55	46.71	0.04	0.00	0.00	0.01	0.00	0.00	0.00	133
20 framboid	P18.1	99.17	51.74	47.33	0.04	0.00	0.03	0.01	0.01	0.00	0.00	132
20 x 80 cleat	P17.2	99.72	52.18	47.40	0.04	0.00	0.01	0.08	0.01	0.00	0.00	130
	P16.2	99.59	52.18	47.29	0.05	0.00	0.02	0.01	0.00	0.01	0.02	128
20 x 40 subhedral	P16.1	99.19	52.08	47.02	0.04	0.00	0.02	0.01	0.00	0.01	0.00	127
	P15.2	98.51	51.58	46.75	0.05	0.00	0.04	0.10	0.00	0.00	0.00	126
30 x 40 subhedral	P15.1	98.19	51.19	46.84	0.05	0.00	0.01	0.10	0.00	0.00	0.00	125
	P14.2	99.88	52.19	47.50	0.05	0.00	0.03	0.08	0.00	0.02	0.00	124
25 x 50 subhedral	P14.1	100.08	52.42	47.55	0.04	0.00	0.01	0.05	0.00	0.00	0.00	123
subhedral	P13.2	99.23	51.83	47.30	0.05	0.00	0.01	0.01	0.00	0.02	0.01	122
30 x 50 irregular/	P13.1	99.21	52.09	47.03	0.05	0.00	0.02	0.00	0.00	0.02	0.00	121
	P12.3	98.39	51.55	46.60	0.06	0.00	0.03	0.16	0.00	0.00	0.00	120
	P12.2	98.64	51.28	47.13	0.04	0.00	0.02	0.17	0.01	0.00	0.00	119
35 x 70 cleat	P12.1	98.35	51.21	46.88	0.06	0.00	0.01	0.18	0.00	0.00	0.00	118
euhedral	P11.2	100.26	52.59	47.58	0.04	0.00	0.04	0.02	0.00	0.00	0.00	117
30 x 50 subhedral/	P11.1	100.17	52.44	47.62	0.04	0.00	0.02	0.04	0.00	0.00	0.00	116
40 x 50 irregular	P10.1	94.20	49.38	44.71	0.05	0.00	0.01	0.03	0.01	0.00	0.00	114
subhedral	P9.3	99.95	52.33	47.54	0.05	0.00	0.02	0.00	0.01	0.00	0.00	113
60 x 70 irregular/	P9.2	98.86	51.64	47.13	0.04	0.00	0.02	0.01	0.01	0.00	0.00	112
25 framboid	P8.4	99.79	51.85	47.72	0.06	0.00	0.03	0.08	0.04	0.01	0.00	110
25 framboid	P8.3	99.06	51.29	47.59	0.05	0.00	0.01	0.05	0.06	0.00	0.00	109
30 framboid	P8.2	98.92	51.47	47.26	0.05	0.00	0.02	0.05	0.05	0.01	0.00	108
60 framboid	P8.1	100.27	51.98	48.13	0.06	0.00	0.01	0.04	0.03	0.01	0.01	107
	P7.2	96.52	50.44	45.99	0.05	0.00	0.01	0.00	0.00	0.03	0.00	106
35 x 40 euhedral	P7.1	98.24	51.13	47.02	0.05	0.00	0.01	0.02	0.00	0.00	0.01	105
	P6.2	100.39	52.40	47.90	0.05	0.00	0.03	0.00	0.00	0.00	0.00	104
25 x 35 subhedral	P6.1	97.95	50.81	47.07	0.04	0.00	0.00	0.02	0.00	0.00	0.00**	103
				eed	31 Flotation Feed	31 F						
Size (microns)/form	Grain	Total	တ	Fe	င္စ	8	Zn	As	<u>z</u>	5	Se	N 0

	161 0.0																			142 0.00		140 0.0					No. Se
																				0 0.03							Cu
0.01	0.03	0.01	0.02	0.00	0.00	0.01	0.02	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.01	0.13	0.07	0.07	0.07	0.00	0.00	0.00	0.00	0.00	٠	Z.
0.02	o o o o o	0.01	0.02	0.03	0.01	0.00	0.02	0.00	0.02	0.03	0.00	0.00	0.01	0.01	0.02	0.06	0.02	0.01	0.00	0.01	0.01	0.00	0.00	0.00	0.00		As
0.02	0.02	0.02	0.02	0.00	0.03	0.01	0.00	0.02	0.02	0.00	0.01	0.02	0.00	0.01	0.03	0.03	0.03	0.03	0.04	0.01	0.02	0.02	0.04	0.02	0.02		Zn
0.00	o o. o. o.	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	03 P	8
0.04	2 0	0	0	0	0	0	0	0.	0	0	0	0.	0.	.0	0	0.	0.	0.	0.0	0.	9	0.05	9	0	9	lant	င္ပ
. هه	2 2	05	8	2	2	2	9	2	4	2	2	8	2	2	2	9	2	05	6	05	95	9	2	2	05	Fee	O _*
4 46.59	-	05 47.78																				05 47.82				Plant Feed	o* Fe
46.59	-	47.78	47.58	44.69	47.00	47.20	45.56	47.92	47.56	45.36	46.13	46.96	46.56	46.60	47.30	47.16	46.90	47.30	47.27	46.90	47.66		47.61	47.85	47.72	Feed	
46.59 50.09	46.98 44.61	47.78 52.03	47.58 51.19	44.69 49.46	47.00 50.68	47.20 51.36	45.56 49.86	47.92 52.28	47.56 52.00	45.36 50.15	46.13 50.49	46.96 51.02	46.56 51.20	46.60 50.46	47.30 50.65	47.16 51.72	46.90 51.72	47.30 51.45	47.27 51.31	46.90 51.38	47.66 50.74	47.82	47.61 51.67	47.85 52.09	47.72 52.08	Feed	Fe
46.59 50.09	46.98 49.74 96.83 44.61 49.50 94.18	47.78 52.03 99.90	47.58 51.19 98.91	44.69 49.46 94.23	47.00 50.68 97.77	47.20 51.36 98.62	45.56 49.86 95.51	47.92 52.28	47.56 52.00 99.66	45.36 50.15 95.58	46.13 50.49 96.69	46.96 51.02 98.05	46.56 51.20	46.60 50.46 97.14	47.30 50.65 98.04	47.16 51.72 99.04	46.90 51.72	47.30 51.45 98.94	47.27 51.31	46.90 51.38	47.66 50.74 98.49	47.82 50.58 98.47	47.61 51.67 99.36	47.85 52.09 100.00	47.72 52.08 99.88	Feed	Fe

	140 141 143	137 138 139	134 135 136	132 133	174 175	171 172	169 170	167 168	165 166	No.
0.00 0.00 0.01	0.00 0.00 0.01	0.00 0.00 0.00	0.00 0.00 0.00	0.00	0.00	0.00	0.00	0.00	0.01	Se 0.01
0.00 0.00 0.00	0.01 0.00 0.00	0.00 0.00 0.00	0.01 0.00 0.00	0.00 0.01	0.05	0.05	0.05	0.07 0.05	0.00	C _u
0.00 0.01 0.00	0.00	0.00 0.00 0.00	0.00 0.00 0.00	0.00	0.07	0.04	0.02	0.03 0.03	0.01	0.01 Z:
0.01 0.03 0.01	0.01 0.00 0.00	0.00 0.00 0.01	0.01 0.00 0.00	0.00	0.00	0.01	0.02	0.01 0.01	0.00	As
0.00 0.00 0.02	0.01 0.02 0.01	0.03 0.02 0.01	0.01 0.00 0.01	0.00	0.02	0.03	0.02	0.01 0.03	0.02	Zn 0.00
0.00 0.00	0.00 0.00	0.00 0.00 0.00	0.00 0.00	51 C 0.00 0.00	0.00	0.00	0.00	0.00	0.00	Cd
0.06 0.04 0.05	0.06 0.06 0.04	0.05 0.05 0.04	0.04 0.05 0.06	51 Cleaned Coal .00	0.05	0.00	0.04	0.04	0.04	Co*
47.90 45.50 46.20	47.52 46.54 46.86	47.7 47.3 46.8	47. 47. 47.	oal 44 46	4 4 4	46	47. 47.	47.: 47.:	46. 47.	Fe 46.7
		22 22 23	06 18	44.84 46.95	46.85 47.09 46.95	.77	95	3 3	28 12	78
51.75 48.56 50.23	51.91 49.81 51.40	72 52.02 32 51.83 32 51.62		.84 48.25 .95 51.59	5.85 51.59 7.09 51.75 5.95 51.80					
			51.81 51.80 51.59			50.82 52.05		51.77 51.93	50.18 52.16	S 51.24
	99.52 96.43 98.34	52.02 99.83 51.83 99.22 51.62 98.51	51.81 51.80 51.59	48.25 51.59	51.75 51.80	50.82 97.75 52.05 100.04	51.26 51.70	51.77 51.93	50.18 96.54 52.16 99.40	S 51.24

No. 151 152	Se 0.00 0.00	Cu 0.05 0.14	0.01 0.00	As 0.04 0.06	Zn 0.00 0.02	0.00 0.00	0.04 0.06	Fe 45.44 45.67	S 49.33 49.59	Total 94.90 95.54	TT TT _	Grain Py8.2 Py9.1
53 54	0.01	0.11	0.02	0.04	0.00	0.00	0.06		44.84 47 77		50.01 51 69	50.01 95.08 51.69 99.56
155	0.00	0.00	0.00	0.03	0.04	0.00	0.06	4	7.84		52.07	52.07 100.03
156	0.00	0.02	0.00	0.00	0.02	0.00	0.05	47	.10		51.23	51.23 98.42
157	0.00	0.00	0.00	0.00	0.01	0.00	0.04	4	7.25		51.25	51.25 98.55
158	0.00	0.00	0.00	0.00	0.02	0.00	0.05		47.70		52.12	52.12 99.90
159	0.01	0.02	0.02	0.01	0.03	0.00	0.06		45.42		49.76	49.76 95.33
161	0.00	0.03	0.03	0.01	0.01	0.00	0.06		46.76		51.89	51.89 98.80
162	0.00	0.01	0.02	0.00	0.02	0.00	0.06		44.77		50.42	50.42 95.31
163	0.00	0.03	0.04	0.02	0.02	0.00	0.06		46.86		51.03	51.03 98.07
164	0.00	0.02	0.00	0.01	0.03	0.00	0.04		46.26		51.99	51.99 98.36
165	0.00	0.06	0.00	0.02	0.02	0.00	0.05		46.33		52.58	52.58 99.07
166	0.00	0.02	0.00	0.02	0.00	0.00	0.05		46.01		52.80	52.80 98.91
					Ę	Flotation Concentrate- Manua	ncentrat	æ	- Manual	- Manual	- Manual	- Manual
167	0.00	0.01	0.00	0.01	0.02	0.00	0.05		46.55		51.98	51.98
168	0.00	0.00	0.00	0.00	0.02	0.00	0.05		45.82		50.11	50.11 96.00
169	0.00	0.00	0.00	0.01	0.00	0.00	0.04		47.10		51.19	51.19 98.35
170	0.00	0.00	0.00	0.00	0.04	0.00	0.04		47.28		51.75	51.75 99.12
171	0.00	0.00	0.00	0.01	0.02	0.00	0.04		47.03		51.67	51.67 98.78
176	0.00	0.00	0.02	0.02	0.02	0.00	0.05		47.04	47.09 51.90 47.04 51.58	51.58	51.58
177	0.00	0.01	0.03	0.04	0.01	0.00	0.07		47.20			51.98 99.34
178	0.00	0.02	0.01	0.02	0.00	0.00	0.04		47.35		52.04	52.04 99.50
179 180	0.00	0.01	0.00	0.01	0.02	0.00	0.04		46.68 47.48	46.68 50.97 47.48 51.42		50.97 51.42
č	0.00	0.00	0.00	0.01	0.00	0.00	0.00		47.48		51.42	51.42 98.96

O	199	198	197	196	195	194	193	192	191	190	189	186	185	184	183	182	181	N _O
0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01	0.00	0.00	0.05	0.00	0.01	0.00	0.00	0.00	0.00	Se
0.01	0.02	0.01	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.01	0.14	0.01	0.00	0.00	0.00	0.01	0.02	δ
0.04	0.10	0.02	0.01	0.00	0.01	0.00	0.00	0.00	0.02	0.00	0.33	0.00	0.00	0.01	0.02	0.02	0.00	<u>z</u>
0.00	0.01	0.02	0.00	0.01	0.01	0.02	0.00	0.00	0.01	0.01	0.01	0.01	0.04	0.00	0.02	0.04	0.02	As
0.01	0.02	0.00	0.02	0.01	0.01	0.02	0.01	0.02	0.04	0.03	0.03	0.01	0.01	0.02	0.02	0.02	0.02	Zn
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	S
0.05	0.04	0.06	0.05	0.04	0.04	0.04	0.05	0.04	0.05	0.05	0.04	0.05	0.05	0.05	0.05	0.04	0.05	ဂ •
47.62	47.50	46.14	46.41	46.56	47.56	47.20	47.64	47.21	46.99	47.54	45.24	46.59	45.21	46.60	47.61	46.92	46.99	Fe
, -	47.50 51.76																	Fe S
, -	51.76	51.04	49.93	49.75	52.10	52.16	51.95	51.37	51.20		51.24	50.50	49.38	51.23	51.40	51.27	51.38	-
51.81	51.76	51.04	49.93	49.75	52.10	52.16	51.95	51.37	51.20	51.63	51.24	50.50	49.38	51.23	51.40 99.12	51.27	51.38	တ

^{*}Co data include 0.03 to 0.05 wt. % contribution of iron interference. **Detection limit for other elements is 0.01 wt. %.

30 x 50 subhedral	p13.1	97.98	52.53	45.34	0.04	0.00	0.02	0.06	0.00	0.00	0.00	46
20 x 70 cleat	p12.1	98.77	52.27	46.41 46.31	0.03	0.00	0.02	0.01	0.01	0.02	0.00	44
20 framboid	p11.1	99.20	52.34	46.75	0.05	0.00	0.01	0.01	0.03	0.00	0.00	43
	p10.2	98.11	51.72	46.30	0.04	0.00	0.02	0.02	0.01	0.00	0.00	42
40 x 50 subhedral	p10.1	97.81	51.79	45.91	0.04	0.00	0.03	0.02	0.01	0.00	0.01	41
	p9.2	97.14	51.30	45.74	0.04	0.00	0.04	0.01	0.00	0.01	0.00	40
25 x 50 framb. cluster	p9.1	96.91	50.98	45.83	0.04	0.00	0.02	0.02	0.01	0.00	0.02	39
	p8.2	97.91	51.68	46.14	0.04	0.00	0.01	0.02	0.00	0.00	0.01	38
25 framboid	p8.1	98.57	51.96	46.54	0.03	0.00	0.01	0.02	0.01	0.00	0.01	37
feathery rim on 7.2	p7.3	94.08	49.43	44.49	0.04	0.00	0.01	0.06	0.02	0.01	0.01	35
80 x 100 composite	p7.2	98.28	52.17	45.98	0.04	0.00	0.02	0.02	0.04	0.00	0.01	34
25 euhedral	p6.1	98.45	51.98	46.39	0.04	0.00	0.01	0.01	0.01	0.01	0.00	32
	p5.2	98.62	51.81	46.72	0.04	0.00	0.03	0.01	0.00	0.00	0.00	3
30 x 40 sub/euhedral	p5.1	97.93	51.66	46.22	0.04	0.00	0.01	0.01	0.00	0.00	0.00	30
	p4.3	99.94	52.48	47.33	0.04	0.00	0.02	0.03	0.03	0.00	0.00	29
	p4.2	98.77	52.24	46.38	0.04	0.00	0.04	0.03	0.03	0.01	0.00	28
70 x 80 composite	p4.1	99.10	51.98	46.98	0.04	0.00	0.03	0.03	0.02	0.00	0.01	27
	p3.3	98.89	52.47	46.18	0.05	0.00	0.03	0.02	0.01	0.14	0.00	26
	p3.2	97.47	51.61	45.70	0.04	0.00	0.03	0.02	0.01	0.05	0.00	25
30 x 70 subhedral	p3.1	98.47	51.67	46.70	0.04	0.00	0.03	0.00	0.00	0.04	0.00	24
	p2.3	98.79	52.67	46.05	0.04	0.00	0.02	0.00	0.00	0.00	0.00	23
	p2.2	99.37	52.30	46.98	0.05	0.00	0.02	0.02	0.00	0.00	0.00	22
50 subhedral	p2.1	98.91	52.59	46.24	0.04	0.00	0.01	0.02	0.00	0.00	0.00	21
	p1.4	97.00	51.28	45.62	0.05	0.00	0.02	0.00	0.01	0.01	0.01	20
	p1.3	97.63	51.40	46.13	0.05	0.00	0.04	0.01	0.00	0.01	0.00	19
	p1.1	97.04	51.33	45.65	0.05	0.00	0.01	0.00	0.01	0.00	0.00	18
50 x 70 composite	p1.1	96.86	50.87	45.93	0.05	0.00	0.00	0.01	0.00	0.00	0.00**	17
				entrate	32 Flotation Concentrate	32 Flotat						
Size(microns)/Form	Grain	Total	တ	Fe	င္	C C	Zn	As	<u>Z</u>	ပ်	Se	N _o

VOX OO SUDIIGUIGII	P4.4	99.56	52.71	46.64	0.03	0.00	0.03	0.01	0.02	0.12	0.00	101
70 v 80 subbodral	P4.2	98.21	51.56 51.06	46.40 46.81	0.02	0.00	0.02	0.03	0.04	0.12	0.02	100
80 subhedral	P4.1	99.45	52.65	46.59	0.03	0.00	0.02	0.01	0.03	0.10	0.00	98
	P3.4	101.11	53.42	47.64	0.03	0.00	0.00	0.01	0.00	0.01	0.00	97
50 framboid	P3.3	99.56	52.74	46.75	0.03	0.00	0.03	0.00	0.00	0.01	0.00	96
	P3.2	99.69	52.66	46.98	0.03	0.00	0.00	0.00	0.00	0.02	0.00	95
50 framboid	P3.1	98.91	51.90	46.93	0.03	0.00	0.04	0.00	0.00	0.01	0.00	94
cluster	P2.4	99.98	52.93	46.93	0.04	0.00	0.02	0.00	0.03	0.04	0.00	93
50 x 60 framboid	P2.3	98.03	52.04	45.84	0.03	0.00	0.01	0.02	0.04	0.03	0.01	92
cluster	P2.2	98.38	52.39	45.86	0.03	0.00	0.02	0.00	0.05	0.01	0.00	91
40 x 60 framboid	P2.1	98.64	52.32	46.15	0.03	0.00	0.03	0.00	0.05	0.05	0.00	90
20 x 25 subhedral	P1.2	100.44	53.00	47.39	0.03	0.00	0.02	0.00	0.00	0.00	0.00	89
20 subhedral	P1.1	100.04	52.87	47.11	0.03	0.00	0.02	0.01	0.00	0.00	0.00	88
				ă	Plant Feed	03						
	P15.3	99.33	52.38	46.84	0.04	0.00	0.03	0.04	0.00	0.00	0.00	87
	P15.2	98.72	52.09	46.54	0.03	0.00	0.04	0.02	0.00	0.00	0.00	86
25 x 150 cleat	P15.1	96.56	51.17	45.30	0.02	0.00	0.03	0.03	0.00	0.01	0.01	85
cluster	P13.2	99.12	52.67	46.38	0.02	0.00	0.04	0.00	0.01	0.00	0.00	83
25 x 30 framboid	P13.1	98.86	52.35	46.46	0.04	0.00	0.01	0.00	0.00	0.00	0.00	82
(framb. cluster?)	P12.2	98.13	51.80	46.27	0.03	0.00	0.03	0.00	0.00	0.00	0.00	81
40 x 80 subhedral	P12.1	99.08	52.09	46.92	0.03	0.00	0.02	0.02	0.01	0.01	0.00	80
	P11.2	97.83	51.30	46.46	0.03	0.00	0.01	0.01	0.00	0.00	0.01	79
30 x 60 subhedral	P11.1	97.31	51.14	46.08	0.04	0.00	0.03	0.02	0.00	0.00	0.00	78
	P10.3	96.56	50.90	45.62	0.03	0.00	0.02	0.00	0.00	0.00	0.00	77
	P10.2	97.10	51.46	45.60	0.02	0.00	0.01	0.01	0.00	0.00	0.00	76
50 x 70 euhedral	P10.1	97.48	51.30	46.10	0.04	0.00	0.02	0.01	0.01	0.00	0.00	75
cluster	P9.2	99.64	52.02	47.55	0.03	0.00	0.01	0.01	0.01	0.02	0.00	74
Size(microns)/Form	Grain	Total	S	Fe	Co*	8	Zn	As	<u>z</u>	င်	Se	N _o

16 17 18 19 19 20 21	N 1 3	3 2 1	10	114	113	3 1 3	109 110	108	106 107	105	104	103	102	N 0
0.00 0.00 0.00 0.00 0.00	0.00 Se	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	Se
0.03 0.02 0.01 0.03 0.03	0.00 Cu	0.00	0.00	0.00	0.00	0.00	0.04	0.00	0.01	0.02	0.02	0.04	0.02	C
0.01 0.01 0.02 0.05 0.05	0.00 N:	0.00	0.00	0.01	0.03	0.00	0.00	0.00	0.01	0.01	0.02	0.03	0.00	<u>z</u>
0.02 0.02 0.01 0.01 0.01	0.00 As	0.01	0.02	0.09	0.00	0.39	0.01	0.00	0.01	0.00	0.00	0.01	0.01	As
0.02 0.00 0.00 0.02 0.02	0.03 Zn	0.00	0.00	0.03	0.02	0.01	0.00	0.02	0.02	0.01	0.02	0.04	0.04	Zn
0.00 0.00 0.00 0.00 0.00	0.00 Cd	0.00	51 C 0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	S
0.05 0.05 0.05 0.06 0.05	0.06 Co*	0.06	51 Cleaned Coal	0.03	0.03	0.04	0.03	0.03	0.03	0.03	0.04	0.03	0.04	C _o *
46.86 46.46 46.97 47.73 46.86 46.98	45.86 47.34 Fe	46.96 46.70	oal 46.86	46.18 46.25	46.27	45.97	46.86 45.57	47.74	47.56 46.81	47.30	46.88	46.75	46.75	Fe e
46.8651.0846.4650.4046.9749.1347.7351.5846.8651.1446.9850.74	40.86 31.34 47.34 51.62 Fe S		16.86		46.56 51.87 46.27 51.69									Fe S
		51.44 51.38	t6.86 51.04	52.48		52.05	51.12 50.40	52.12	51.80 51.80	52.14	52.99	52.16	52.30	
51.08 50.40 49.13 51.58 51.14 50.74	51.62 99.05 S Total	51.44 51.38	1 6.86 51.04 97.98	52.48	51.87 99.06 51.69 98.04	52.05 98.46	51.12 50.40	52.12 99.92	51.80 51.80	52.14 99.51	52.99	52.16	52.30	Ø

Ċη	44	43	42	41	40	39	38	37	36	35	34	33	32	31	30	23	22	N _O
0.01	0.00	0.00	0.02	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	Se
0.11	0.05	0.00	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.01	င်
0.00	0.00	0.05	0.07	0.03	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.02	0.00	0.01	0.02	<u>z</u>
0.00	0.01	0.01	0.02	0.02	0.01	0.01	0.02	0.01	0.01	0.00	0.01	0.01	0.01	0.00	0.02	0.02	0.00	As
0.01	0.02	0.01	0.01	0.00	0.00	0.02	0.01	0.02	0.02	0.00	0.02	0.02	0.01	0.01	0.00	0.02	0.03	Zn
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	C
0.05	0.05	0.06	0.06	0.05	0.06	0.06	0.05	0.06	0.06	0.06	0.05	0.05	0.06	0.06	0.05	0.06	0.05	င္ပ
47.18	47.61	48.14	47.83	47.61	47.10	47.23	46.99	47.56	47.99	47.84	48.36	47.18	47.12	47.74	48.17	48.27	46.62	Fe
-	47.61 51.62		-			-			_			-				•		Fe S
50.41		51.76	51.56	51.19	50.78	50.79	50.31	50.72	51.72	51.73	51.75	51.35	51.32	51.93	51.69	52.54	50.82	
50.41	51.62 99.36	51.76	51.56	51.19	50.78	50.79	50.31	50.72	51.72	51.73	51.75 100.20	51.35 98.62	51.32	51.93 99.76	51.69	52.54	50.82	တ

^{*}Co data include 0.03 to 0.05 wt. % contribution of iron interference. **Detection limit for other elements is 0.01 wt. %.

40 x 70 composite	Py15.1	97.14	50.62	46.30	0.07	0.00	0.00	0.15	0.00	0.01	0.00	39
20 euhedral	Py14.1	97.12	50.64	46.41	0.06	0.00	0.00	0.01	0.00	0.01	0.00	38
	Py13.3	96.21	50.10	46.01	0.05	0.00	0.00	0.04	0.00	0.00	0.00	37
	Py13.2	96.44	50.35	46.00	0.06	0.00	0.00	0.03	0.00	0.00	0.00	36
35 x 100 subhedral	Py13.1	96.38	50.00	46.27	0.06	0.00	0.00	0.04	0.00	0.01	0.00	35
	Py12.2	95.28	49.67	45.48	0.06	0.00	0.00	0.07	0.00	0.01	0.00	34
25 x 25 subhedral	Py12.1	95.18	49.74	45.35	0.05	0.00	0.00	0.03	0.01	0.00	0.00	33
	Py11.2	96.83	50.50	46.26	0.05	0.00	0.00	0.00	0.01	0.00	0.00	32
30 x 120 subhedral	Py11.1	96.79	50.52	46.09	0.06	0.00	0.00	0.09	0.00	0.02	0.01	31
	Py10.2	96.05	50.63	45.34	0.06	0.00	0.00	0.00	0.00	0.02	0.00	30
20 x30 subhedral	Py10.1	96.78	50.91	45.81	0.05	0.00	0.00	0.00	0.01	0.00	0.00	29
	Py9.3	98.58	51.70	46.81	0.07	0.00	0.00	0.00	0.00	0.00	0.00	28
	Py9.2	94.97	49.52	45.22	0.07	0.00	0.01	0.08	0.02	0.05	0.01	27
50 x 50 subhedral	Py9.1	97.56	51.31	46.13	0.07	0.00	0.00	0.02	0.00	0.02	0.00	26
	Py8.3	95.79	50.43	45.27	0.06	0.00	0.00	0.00	0.01	0.01	0.01	25
	Py8.2	96.14	51.01	45.02	0.07	0.00	0.01	0.02	0.00	0.00	0.01	24
60 x 70 subhedral	Py8.1	96.65	50.48	46.00	0.07	0.00	0.01	0.02	0.01	0.05	0.00	23
30 x 30 subhedral	Py7.4	98.42	51.98	46.32	0.06	0.00	0.00	0.00	0.04	0.01	0.00	22
	Py7.3	97.54	51.77	45.63	0.06	0.00	0.00	0.00	0.05	0.02	0.01	21
	Py7.2	98.05	51.34	46.61	0.06	0.00	0.00	0.00	0.04	0.01	0.00	20
50 x 80 irregular	Py7.1	96.50	50.82	45.57	0.05	0.00	0.00	0.00	0.05	0.00	0.00	19
12 euhedral in cluster	Py6.1	98.66	52.44	46.03	0.06	0.00	0.00	0.10	0.02	0.00	0.00	18
8 euhedral in cluster	Py5.1	97.74	51.56	45.94	0.07	0.00	0.00	0.16	0.01	0.00	0.00	17
10 euhedral	Py4.1	98.83	52.57	46.09	0.07	0.00	0.00	0.08	0.02	0.00	0.00	16
	Py2.2	96.71	50.83	45.71	0.05	0.00	0.01	0.12	0.00	0.00	0.00	12
25 round/euhedral	Py2.1	97.12	51.17	45.79	0.05	0.00	0.00	0.09	0.00	0.00	0.02	11
	Py1.2	95.32	50.19	44.99	0.06	0.00	0.00	0.03	0.01	0.04	0.00	10
20 euhedral	Py1.1	98.41	51.81	46.47	0.07	0.00	0.00	0.01	0.01	0.05	0.00**	9
				ă.	Plant Feed	03						
•												
Size (microns)/Form	Grain	Total	တ	Fe	င္ပ	S C	Zn	As	<u>z</u>	ပ	Se	N _o

	0 0 0 0	63 60	56 57	54 54	52	50	49	47	4 6		45	t 4	2 2	4	40	N 0
0.00 0.00 0.01	0.10	0.00 0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	Se
0.01 0.11 0.16	0.11	0.00 0.01 0.07	0.00	0.00	0.02	0.01	0.02	0.00	0.01		0.01	0.02	0.02	0.01	0.00	5
0.00	0.15	0.01 0.00	0.02	0.03	0.11	0.09	0.07	0.04	0.07		0.02	0.02	0.00	0.00	0.00	<u>Z</u>
0.27 0.37 0.02 0.18	0.13	0.04 0.04	0.15 0.04	0.25 0.08	0.83	0.60	0.54	0.19	0.35		0.03	0.03	0.20	0.04	0.17	As
0.01 0.00	0.00	0.01 0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	Zn
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	51 CI	0.00	0.00	0.00	0.00	0.00	8
0.06 0.06 0.06	0.22	0.06 0.06	0.06	0.07	0.06	0.05	0.07	0.06	0.06	51 Cleaned Coal	0.06	0.05	0.05	0.08	0.08	Co _*
										\sim						
45.34 45.07 45.14	46.25	45.94 45.51 44 70	45.52 46.11	45.44 46.29	46.16	46.24 45.56	46.65	45.71 45.78	46.32	Soal	46.22	46.02	45.75 46.22	45.76	45.54	Fe
	46.25 51.62 46.05 50.65									Soal		46.02 50.50				Fe S
	51.62 50.65	50.53 50.64 49.53	50.48 51.35	50.41 50.13	51.16	51.07 50 60	50.90	49.42 50 54	50.11	Soal	50.51		50.64	50.62	50.48	
50.27 50.03 50.82 49.32	51.62 98.59 50.65 97.24	50.53 96.58 50.64 96.28 49.53 94.62	50.48 96.24 51.35 97.56	50.41 50.13	51.16 98.33	51.07 50 60	50.90 98.25	49.42 50 54	50.11	Soal	50.51 96.85	50.50	50.64 96.66	50.62 96.50	50.48	တ

0.02 0.02 0.15 0.02 0.00 0.05 0.01 0.01 0.01 0.02 0.00 0.05 0.02 0.02 0.02 0.01 0.00 0.05	0.02 0.02 0.03 0.00 0.00 0.06 0.02 0.01 0.10 0.02 0.00 0.05	0.01 0.02 0.02 0.00 0.05 0.00 0.03 0.02 0.00 0.05	0.01 0.01 0.04 0.03 0.00 0.05 0.00 0.02 0.03 0.03 0.00 0.05	0.00 0.00 0.04 0.02 0.00 0.05 0.07 0.01 0.03 0.03 0.00 0.04	0.02 0.00 0.08 0.01 0.00 0.05	32 Flotation Concentrate	0.00 0.00 0.02 0.01 0.00 0.05	0.02 0.00 0.09 0.00 0.00 0.07	0.01 0.00 0.05 0.00 0.00 0.06	0.00 0.01 0.00 0.00 0.00 0.00 0.06	0.01 0.01 0.02 0.00 0.00 0.00 0.07	0.00 0.02 0.00 0.00 0.00 0.06	0.00 0.00 0.00 0.00 0.01 0.00 0.07	0.01 0.08 0.09 0.06 0.00 0.00 0.07	0.00 0.07 0.02 0.04 0.00 0.00 0.06	0.00 0.00 0.01 0.14 0.00 0.00 0.06	31 Flotation Feed	(
						ယ္												70
0.00 0.00 0.00	0.00	0.00	0.00	0.00	0.00	2 Flotatio	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	31 Flo	G
0.05 0.05 0.05	0.06 0.05	0.05	0.05 0.05	0.05	0.05	n Conce	0.05	0.07	0.06	0.06	0.07	0.06	0.07	0.07	0.06	0.06	tation Fe	Ç
47.13 47.81 47.30	45.34 47.03	47.35 46.90	48.22 47.49	47.71 43.82	47.58	entrate	45.84	45.90	46.40	45.73	45.77 45.72	45.75	46.41	45.46	45.54	45.40	ed.	T O
~																		
	48.66 50.89	51.48 51.15	51.85 50.83	51.34 50.90	50.71		50.92	50.63	50.47	50.49	49.61 50.88	49.02	50.73	50.01	50.06	50.86		U
51.28 51.31 51.29				51.34 99.15 50.90 94.92				50.63 96.70			49.61 95.48 50.88 96.74							o i otal
51.28 51.31 51.29		98.98 98.16	100.20 98.46		98.46			96.70 96.84	97.00	96.28	95.48 96.74	94.85	97.22		95.80	96.46 96.53		

25 x 40 subhedral	P11.2	98.51	51.28	47.03	0.05	0.00	0.02	0.06	0.00	0.05	0.01	72
	P2.4	97.70	50.90	46.58	0.06	0.00	0.03	0.07	0.02	0.02	0.03	70
	P2.3	98.99	51.57	47.12	0.05	0.00	0.00	0.05	0.01	0.14	0.05	69
	P2.2	98.89	51.39	47.30	0.05	0.00	0.00	0.08	0.01	0.02	0.03	68
25 framboid	P2.1	98.27	50.92	47.23	0.06	0.00	0.00	0.04	0.00	0.02	0.00	67
	P3.3	98.07	51.01	46.86	0.05	0.00	0.00	0.07	0.00	0.07	0.00	66
	P3.2	98.52	51.24	47.08	0.05	0.00	0.02	0.06	0.01	0.05	0.00	65
40 euhedral	P3.1	98.72	51.12	47.40	0.05	0.00	0.01	0.06	0.00	0.09	0.00	62
	P10.2	99.09	51.63	47.37	0.05	0.00	0.01	0.00	0.01	0.02	0.01	63
30 x 50 subhedral	P10.1	99.02	51.40	47.53	0.05	0.00	0.00	0.02	0.00	0.01	0.00	62
cell-fill	P9.2	99.24	51.84	47.20	0.06	0.00	0.02	0.00	0.06	0.07	0.00	61
40 framboid or	P9.1	98.47	51.27	47.09	0.05	0.00	0.02	0.01	0.01	0.02	0.00	60
Size (IIIICIOIIS)/FOITI	<u>G</u>	ו	O	ď	ξ	2	1	ð	2	Ç	ď	Ž.
Cito (misrons)/Eorm)]	Total	n	П)	3	75	٥	Z	2	n N	<u>z</u>

^{**}Detection limit for other elements is 0.01 wt. %. * Co data include 0.05% (32 Flotation Concentrate) to 0.06% (other samples) contribution of iron interference.

B. Illite Analysis

03 Plant Feed Series

8 33 33 35 35 35 35 24 25 27 30 10 12 13 15 8 AI203 28.99 28.59 24.27 27.10 34.42 33.53 27.23 32.69 27.66 33.20 28.72 34.42 34.79 22.13 29.26 CaO 0.48 0.03 0.01 0.01 0.00 0.04 5.24 0.55 0.08 0.17 0.17 0.18 0.16 0.25 0.20 Na20 0.39 0.31 0.10 0.55 0.35 0.94 0.45 0.40 0.33 0.21 0.05 0.14 0.12 0.16 0.24 MgO 4.48 0.55 2.13 0.90 0.63 1.22 0.54 0.62 0.38 0.86 1.38 1.72 1.07 1.68 Cr203 0.02 0.03 0.01 0.01 0.02 0.03 0.02 0.03 0.02 0.01 0.00 0.02 MnO Pittsburgh Coal (PA) 0.06 0.01 0.03 0.03 0.10 0.03 0.00 0.02 0,00 0.00 0.00 0.00 0.00 0.03 FeO 7.51 1.72 6.47 2.07 3.22 0.98 4.731.791.19 2.52 0.84 3.32 0.97 **天20** 5.34 5.66 5.73 5.45 2.75 0.05 4.62 4.12 2.50 4.11 2.34 2.11 1.28 1.29 59.60 47.29 SiO2 52.76 63.77 45.03 51.17 52.56 49.43 60.87 50.81 60.03 45.09 54.04 62.24 50.26 TiO2 0.33 6.98 0.30 0.02 1.79 0.14 0.17 1.20 0.80 0.14 1.06 0.31 0.26 0.13 0.28 Total 94.20 97.98 91.08 90.90 92.62 90.00 91.77 92.38 95.81 96.15 89.80 93.80 92.69 90.15 88.91 Comment illitel3.3 illite 1.5 illite 1.4 illite3.5 illite4.4 illite4.3 illite4.2 illite4.1 illite3.8 illite3.2 illite2.2 illite1.7 illite1.3 illite1.2

40 43

26.03 25.69

3.47

0.96

0.02

0.02 0.01

2.18 4.61

60.86 52.60

0.12 0.12

98.53 96.80

90.31

illite5.6 illite5.4 illite5.1

2.40

1.41

0.03

2.69

58.77

0.45

illite4.7

1.65

31.84

2.30 3.06 0.76

0.19

0.57

0.01

0.00

1.02 1.81

1.65

78 79	73 74 75 76	69 70 71 72	63 64 67	56 57 58	48 49 51 52 55	N _o
27.61 32.47	36.38 31.95 34.30 35.85 34.73	33.09 33.29 24.71 33.91	21.72 33.61 32.70 29.93 27.65	37.83 27.46 34.43 35.87	33.54 32.63 32.57 34.13 33.38 32.76	AI203
0.18 0.20	0.10 0.18 0.15 0.16 0.16	0.22 0.19 0.16 0.15	0.08 0.02 0.11 0.11	0.08 0.05 0.07 0.06	0.08 0.17 0.16 0.17 0.12 0.13	CaO
0.25 0.12	0.16 0.28 0.31 0.27 0.25	0.34 0.34 0.26 0.30	0.16 0.33 0.28 0.22 0.23	0.10 0.15 0.09 0.38	0.30 0.44 0.34 0.10 0.22 0.31	Na2O
1.58 1.66	0.52 1.11 1.11 1.16 0.92	1.37 1.14 0.96 0.99	1.06 1.27 1.78 1.27 2.29	0.28 1.44 1.60 1.33	1.25 1.44 1.56 1.51 1.03	MgO
0.02	0.01 0.04 0.04 0.03 0.03	0.04 0.02 0.02 0.03	0.01 0.02 0.00 0.03 0.01	0.00 0.02 0.04 0.03	0.02 0.04 0.03 0.01 0.01	Cr203
0.04	0.01 0.00 0.00 0.02	0.00 0.02 0.01 0.03	0.02 0.01 0.00 0.02 0.03	0.01 0.01 0.04 0.00	0.02 0.00 0.02 0.02 0.02 0.02	MnO Kittanning
4.15 4.39	0.95 2.46 2.31 2.50 1.94	3.02 2.48 2.12 1.94	2.44 3.09 3.24 2.91 4.59	0.92 2.86 5.66 2.73	1.98 2.49 2.69 2.66 2.18 1.89	MnO FeO K2O Kittanning Coal Zone (PA)
3.39 3.20	1.39 2.46 2.56 1.87 2.11	2.88 2.70 1.97 2.17	3.63 5.99 3.65 5.48 2.66	0.37 3.18 0.68 3.51	3.17 3.42 3.35 1.31 2.93 3.00	K2O ne (PA)
54.31 52.07	51.27 49.07 53.30 51.13 54.01	48.64 49.33 64.74 53.72	63.83 50.97 49.83 51.38 61.08	50.04 59.46 49.70 49.73	49.21 49.60 50.71 53.37 48.84 48.35	SiO2
0.27 0.29	0.30 0.00 0.62 0.54 0.55	0.87 0.65 0.51 0.54	0.62 0.58 0.15 2.73 0.16	0.04 0.18 0.16 0.16	1.22 1.61 0.81 0.72 0.38 0.82	TiO2
91.80 94.44	91.12 87.54 94.70 93.53 94.68	90.46 90.16 95.46 93.78	93.57 95.89 91.74 94.08 98.83	89.69 94.83 92.46 93.80	90.78 91.83 92.23 93.99 89.08 88.36	Total
illite5.1 illite5.2	illite4.5 illite4.6 illite4.7 illite4.8 illite4.9	illite4.1 illite4.2 illite4.3 illite4.4	illite3.3 illite3.5 illite3.6 illite3.9 illite3.2	illite2.1 illite2.2 illite2.3 illite2.4	Illite 1.1 Illite 1.2 Illite 1.4 Illite 1.5 Illite 1.7 Illite 1.7	Comment

123 124	120		115	112	111	108	106	105	100	99	98	97		87	83	81		No.
30.88 25.89	19.17		19.22	27.27	32.34	28.79	15.61	31.70	28.59	17.85	19.10	17.80		26.74	23.78	26.02		AI203
0.2 4 0.23	0.31		0.20	0.25	0.05	0.33	0.20	0.22	0.06	0.15	0.18	0.23		0.08	0.06	0.12		CaO
0.21 0.25	0.44		0.14	0.04	0.14	0.09	0.10	0.13	0.35	0.07	0.04	0.17		0.16	0.11	0.21		Na20
0.76 0.73	0.44		0.98	0.75	2.16	1.04	2.78	0.41	1.80	0.51	0.21	0.70		0.70	1.02	4.55		MgO
0.01 0.02	0.01	_	0.02	0.00	0.00	0.00	0.01	0.00	0.03	0.00	0.01	0.03		0.02	0.02	0.02		Cr203
0.00 0.02	0.02	Pittsburgh	0.00	0.01	0.05	0.00	0.09	0.01	0.00	0.00	0.00	0.01	Freeport (0.00	0.02	0.04	Kittanning	MnO
0.92 1.32	0.45	Pittsburgh Coal (WV)	1.76	1.63	4.25	1.28	8.68	1.52	2.96	1.23	0.82	1.80	Freeport Coal Zone (PA)	1.83	2.48	10.05	Kittanning Coal Zone (PA)	FeO
1.32 2.43	1.63	B	2.51	2.21	5.94	2.59	0.90	1.06	7.79	2.80	1.70	2.33	e (PA)	2.18	4.26	3.01	ne (PA)	X 20
58.85 63.95	71.75		68.50	52.88	47.12	55.54	63.65	54.14	57.35	58.10	60.87	60.28		56.08	55.52	55.00		SiO2
0.30 0.37	0.26		0.14	0.30	0.24	0.17	0.38	0.12	0.43	0.16	0.18	0.52		0.11	0.14	0.24		TiO2
93.48 95.21	94.48		93.48	85.34	92.28	89.84	92.42	89.30	99.36	80.87	83.12	83.88		87.89	87.41	99.27		Total
illite2.2 illite2.3	illite1.2		illite6.2	illite5.4	illite5.3	illite4.5	illite4.3	illite4.2	illite3.2	illite3.1	illite2.4	illite2.3		illite6.4	illite5.6	illite5.4		Comment

132 133 2			128 2				No.
31.88 27.12	21.62	30.38	24.48	9.34	30.45		AI2O3
0.33 0.46	0.31	0.36	0.20	0.29	0.40		CaO
0.41 0.30	0.34	1.05	0.68	1.09	0.94		Na20
0.88 1.34	0.93	1.21	0.76	1.44	1.19		MgO
0.03 0.01	0.02	0.04	0.01	0.03	0.02	_	Cr203
0.00 0.03	0.00	0.01	0.01	0.02	0.02	Pittsburgh	MnO
0.86 1.49	0.85	0.93	0.77	1.21	1.25	Pittsburgh Coal (WV)	FeO
5.40 3.64	3.06	3.33	2.98	3.77	3.97	Ķ	K20
56.89 63.87	65.26	55.93	69.56	60.31	50.91		SiO2
0.65 0.55	0.75	0.62	0.23	1.23	2.22		TiO2
97.32 98.81	93.14	93.86	99.67	98.72	91.35		Total
illite4.2 illite4.3	illite4.1	illite3.5	illite3.3	illite3.2	illite3.1		Comment

Appendix III

Elemental Analysis -Quality Control

Elemental Analysis— Quality Control

All of the samples were analyzed by several techniques. Table 2 shows the data for the un-leached feed coals and intermediate and final coal cleaning products of the four program coals. Elemental compositions of all 16 samples were determined by instrumental neutron activation analysis (INAA), inductively coupled plasma - atomic emission spectroscopy (ICP-AES), and inductively coupled plasma - mass spectrometry (ICP-MS). All samples were also analyzed by cold vapor atomic absorption (CVAA) for mercury and hydride generation atomic absorption (HGAA) for selenium. Comparison of results from different independent procedures can provide information on reliability of data analysis and sampling. There is generally very good agreement between values for the same element measured by different techniques, with the exception of the ICP-MS values for arsenic which seems to be biased toward high values. In addition, Table 2 shows the results of duplicate INAA analyses of several program samples. Agreement between these replicates is excellent.

With each group of about 30 samples, the USGS standard coal CLB-1 was analyzed by INAA as a control sample. Table A3-1 shows eight different analyses of CLB-1 by INAA. The mean and standard deviation of the values, and the mean of the reported errors are also given together with the accepted values. There is excellent agreement between the measured values and the "accepted" values. Five control standards were used for ICP-AES. Determined values and accepted values are given in Table A3-2. Several control samples were used for ICP-MS. These control samples, along with accepted values, are given in Table A3-3. Agreement between measured values and accepted values for the ICP techniques is generally good, again with the exception of arsenic, which seems to be biased towards higher values relative to the accepted values for the whole coal control samples. As a result, greater consideration was given to the INAA values for arsenic. In addition, several control samples were determined by cold-vapor atomic absorption (Hg) and by hydride generation atomic absorption (Se). These control samples are included in Table A3-4.

Table A3-1. Control Samples- INAA

Accepted Value: Error:	Mean: Standard Deviation: Mean Error:	CLB Split 9 ERROR: CLB Split 10 ERROR:	ERROR: CLB Split 8 ERROR:	ERROR: CLB Split 6 ERROR:	CLB Split 4 ERROR: CLB Split 5	CLB Split 2 ERROR: CLB Split 3	units: CLB Split 1 ERROR:	
₹ 13	14.0 0.60 0.44	14.4 0.50 14.2 0.46	0.49 13.4 0.41	0.40 15.2 0.49	13.7 0.41 13.7	14.1 0.42 14.4	ppm 13.2 0.39	>
							ND DP m	<u>_</u>
9.7 1.2	10 1.9 0.52	15.7 0.63 10.0 0.68	0.39 9.4 0.39	0.69 10.2 0.37	9.8 0.49 9.2	10.1 0.60 9.6	9.5 0.61	?
₹ 2	2.0 0.21 0.20	2.1 0.20 2.3 0.25	0.32 2.1 0.18	0.15 2.4 0.16	2.0 0.25 2.0	1.9 0.16 1.9	9pm 1.8 0.13)
18 2	19 2.3 3.2	3.3 21 4.3	3.8 19 2.7	2.6 21 2.7	20 4.2 18	17 3.7 19	ppm 15 2.3	<u>?</u>
7 0.7	7.1 0.39 0.21	6.6 0.31 7.0 0.15	0.15 7.0 0.14	0.17 8.1 0.17	6.8 0.42 7.4	7.0 0.19 7.0	ppm 7.1 0.23	}
7.5	1.53 0.038 0.057	1.49 0.059 1.50 0.045	0.072 1.57 0.045	0.082 1.46 0.053	1.57 0.050 1.53	1.51 0.048 1.52	9pm 1.56 0.042	5
						3888	N N D B	<u> </u>
						3888	N N D C)
48 4	50 2.5 3.2	51 51 2.2	2.9 46 4.0	2.2 3.8 3.8	49 3.1 52	48 2.2 50	2n ppm 47 2.8	7
						3 8 8 8 5 8 8 8	ND Ppm	J
					8888		N N N	.
₹.5	1.40 0.051 0.070	1.37 0.046 1.44 0.042	0.040 0.040 1.37 0.041	0.10 1.51 0.097	1.3 0.13 1.4	1.38 0.077 1.37	ppm 1.35 0.041	ļ
0.55 N	0.52 0.058 0.067	0.61 0.077 0.56 0.076	0.075 0.45 0.061	0.064 0.51 0.060	0.45 0.062 0.59	0.49 0.064 0.52	ppm 0.47 0.063	=
1.1 0.44	0.89 0.014 0.019	0.90 0.017 0.89 0.020	0.016 0.88 0.020	0.016 0.93 0.023	0.89 0.021 0.89	0.90 0.021 0.89	0.88 0.016	ָר י

ND = not determined IV = informational value

Table A3-2 Control Samples- ICP-AES

NIST 1633b Accepted Value (7)	NIST 1633a Accepted Value (6)	NIST 1632b (4) Accepted Value (5)	CLB-1 Split 1 (2) CLB-1 Split 2 (2) Mean Accepted Value (3)	Ref 65-1 Ref 65-2 Ref 65-3 Ref 65-4 Ref 65-5 Mean Standard Deviation Accepted Value (1)	Element units:
N D	ND	ND	N N	88888	As ppm
N D	N D	N D	N N		Hg
210 198.2	200 196	= =	9.2 8.8 9.0 9.7	0.024 0.014 0.024 0.018 0.018 0.026 0.021 0.0050 0.019	Cr
N D	N D	N D	N N	N N N N N	Se ppm
130 120.6	130 127	6.7 6.10	17 17 17 18	88888	ppm <u>N</u>
54 50	46 46	2.3 2.29	6.4 6.2 7.0		Co
N D	N	N D	<u> </u>		Sb
N D	ND	ND	N N	NNNN	Pb
ND	N D	N D	N N		ppm Cd
220 210	220 220	11.3 11.89	46 46 48	0.34 0.46 0.62 0.61 0.67 0.54 0.14	Zn
NR NR	12	0.6 NR	0.9 N 1	0.005 0.004 0.006 0.006 0.007 0.007 0.006 0.0011 0.185	Be ppm
140 131.8	180 190	12.0 12.4	8.0 7.8 7.9 8	0.28 0.25 0.34 0.33 0.36 0.36 0.042 0.042	Mn
28 25.7	25 24.7	1.3 1.342	11.4	88888	Th
N D	N D	N D	N N	88888	ppm U
ND	N	0.703 0.759	0.756 0.752 0.754 1.25	0.62 0.50 0.90 1.0 0.80 0.76 0.20	% F

⁽¹⁾ U.S. Geological Survey Water Standard Certificate of Analysis

⁽²⁾ Values based on acid digest of CLB-1 ash recalculated to a whole coal basis

⁽³⁾ U.S. Geological Survey Certificate of Anaysis

⁽⁴⁾ Values based on dissolution of NIST 1632b coal ash reported on a whole coal basis (5) National Bureau of Standards Certificate of Analysis for 1632b

⁽⁶⁾ National Bureau of Standards Certificate of Analysis for 1633a

⁽⁷⁾ National Bureau of Standards Certificate of Analysis for 1633b

ND = Not determined

NR = Not reported

Table A3-3. Control Samples- ICP-MS

T-105 T-105 T-105 Mean Standard Deviation Accepted Value (8)	T-131 T-131 T-131 Mean Mean Standard Deviation Accepted Value (7)	CLB-1 Split 1 (5) CLB-1 Split 2 (5) Mean Accepted Value (6)	NIST 1633b Accepted Value (4)	NIST 1633a Accepted Value (3)	NIST 1632b (1) Accepted Value (2)	Element units:
3.0 2.6 2.5 2.7 0.28 2.3	49 51 51 50 1.0 56.6	16 17 17 13	160 136.2	180 145	4.8 3.72	As ppm
NNN	N N N	N N	N D	N D	N	ppm Hg
888	888	N N	S	N D	N D	Cr
888	888	N N	N D	N D	N	Se
888	888	N N	N D	N D	Z D	ppm <u>Ni</u>
NNN	NNN	N N	N D	N D	S	ppm Co
5.5 4.9 5.0 5.1 0.32	57 55 57 56 1.2 56.2	1.6	6.4 6	7.5 7	0.27 0.24	Sb
11.6 12.2 12.2 12.0 12.0 0.34 11.0	19.9 19.7 19.5 19.7 0.20 18.1	5.5 5.5 5	78 68.2	77 72.4	4.5 3.67	Pb ppm
2.8 2.7 2.7 2.8 0.07	6.8 6.5 6.7 6.7 0.15 6.1	0.096 0.096 0.096 NR	0.92 0.784	1.3	0.071 0.0573	ppm Cd
888	888	N N	N D	N D	N D	Zn
8 8 8 8 8 8	8 8 8 8	Z Z D D	N D	N D	N D	Be ppm
888	888	N N	ND	<u>N</u>	N D	Mn Mn
888	. 888	<u>Z</u> Z	N D	N D	N D	ppm Th
0.4 0.4 0.0 R	8 8 8	0.49 0.47 0.48 0.55	9.5 8.79	11 10.2	0.46 0.436	ppm U
NNN	N N N	Z Z D D	N D	N D	N D	% Fe

NR = Not reported

⁽¹⁾ National Bureau of Standards Certificate of Analysis for 1632b (2) Values based on dissolution of NIST 1632b coal ash reported on a whole coal basis

⁽³⁾ National Bureau of Standards Certificate of Analysis for 1633a

⁽⁴⁾ National Bureau of Standards Certificate of Analysis for 1633b

⁽⁵⁾ Values based on acid digest of CLB-1 ash recalculated to a whole coal basis
(6) U.S. Geological Survey Certificate of Anaysis
(7) U.S. Geological Survey Internal Water Standard Certificate of Anaysis
(8) U.S. Geological Survey Internal Water Standard Certificate of Anaysis

ND = Not determined

Table A3-4 Control Samples-Cold Vapor (Hg) and Hydride Generation (Se) Atomic Absorption

Element . units:	Hg ppm	Se ppm
CLB-1 CLB-1 CLB-1 CLB-1	0.12 0.12 0.13 0.17 0.15	2.86 2.85 1.77
CLB-1 Mean Standard Deviation Accepted Value (1)	0.16 0.14 0.021 0.2	2.49 0.626 2
1632b 1632b 1632b 1632b 1632b 1632b	0.05 0.06 0.05 0.04 0.03 0.04	1.24 0.96 1.19
1632b Mean Standard Deviation Accepted Value (2)	0.05 0.046 0.0092 NR	1.13 0.15 1.29
1633b 1633b 1633b 1633b 1633b 1633b 1633b Mean Standard Deviation Accepted Value (3)	0.15 0.16 0.17 0.15 0.14 0.14 0.12 0.15 0.15 0.15 0.15	ND ND ND ND ND ND ND
1635 1635 1635 Mean Standard Deviation Accepted Value (4)	ND ND ND	0.81 0.74 0.86 0.80 0.060 0.9

⁽¹⁾ U.S. Geological Survey Certificate of Anaysis

NR = not reported

ND = not determined

⁽²⁾ Values taken from National Bureau of Standards Certificate of Analysis for 1632b

⁽³⁾ Values taken from National Bureau of Standards Certificate of Analysis for 1633b

⁽⁴⁾ Values taken from National Bureau of Standards Certificate of Analysis for 1635